Chemical & Process Engineering

CONTENTS

Topics of the Month

Food engineering progress

AN example of how basic research can have immediate practical applications is given in the Report of the D.S.I.R.'s Food Investigation Board for 1953 (H.M.S.O., 3s. 6d.). Arising directly out of biochemical work at the Low Temperature Research Station, a pilot plant on a semi-technical scale, for the isolation of ascorbic acid from plant extracts was designed. The Board's interest in this matter arose from the fact that ascorbic acid can be used as an improver for flour used in bread-making. Trials were carried out jointly with the National Research Development Corporation, who installed the apparatus. In 1953 the process was operated with a hot water extract of lucerne during the crop season. The product obtained was a concentrated syrup which contained ascorbic acid and malic acid substantially free from other plant extractives. The average overall yield of ascorbic acid was nearly 60% of that present in the original extract. Experience in operating the pilot plant established the feasibility of the process, but the estimated cost of full-scale production was about the same as for the production of synthetic crystalline ascorbic acid. Ways of reducing the cost of the process are being sought.

Another promising field of investigation is in the development of plant for freezing fish at sea. Ice is an inadequate preservative for the whole catch of vessels fishing in distant waters and quick freezing and low-temperature cold storage are the only known methods of preserving quality over long periods. A freezer which is stated to be more suitable than existing types for freezing gutted unfilleted fish has been designed and tested on shore and on the Torry Research Station's vessel *Keelby* during the past three years. This experimental plant the Torry vertical plate freezer, is now being developed commercially. In the present design, direct expansion of the primary refrigerant, *Arcton* 6, is used, but with forced flooded circulation higher rates of freezing could possibly be achieved and the pipework involved would be simpler. Tests of a circuit incorporating this system have begun, using a single-station vertical plate freezer, with *Arcton* 4 as the refrigerant. The two successful runs so far accomplished seem to indicate that the performance of this system may be an improvement on that of the direct expansion system. It has been difficult to find a suitable seal for the shaft of the pump circulating the low-temperature liquid refrigerant. Sealing the motor hermetically into the pump casing raises difficulties of electrical supply in existing trawlers. The best mechanical seal used so far withstood 170 hours of severe running before failure, but, with the improved types of seal now available, the running life should be increased.

Atomic power engineering

THE important part that chemical engineering has to play in the development of atomic energy for industrial purposes has already been demonstrated in the work that has been done on atomic projects here and abroad. Since the most immediate industrial application of atomic energy is in the production of electric power, its impact on electrical engineering, too, is considerable. In a paper presented to the British Electrical Power Convention at Eastbourne recently, Sir George Nelson, chairman and managing director of the English Electric Co., summed up the situation from the electrical engineering angle. He said the engineer and the industry must learn to use and handle new materials, to add liquid sodium and heavy water and a host of others to copper and iron as ordinary engineering materials, and must learn to think of neutron economy as familiarly as of power factor. A new engineering will be created within electrical engineering.

Discussing the Sellafield power station Sir George Nelson pointed out that this prototype atomic power station will serve as a training ground for those commercial firms who will take over the design and construction of further nuclear power stations, and so fit themselves not only to supply the British Electricity Authority but also other countries with atomic power plant.

Sulphur recovery in the textile industry

METHODS of recovering sulphur used, mainly in the form of carbon disulphide and sulphuric acid, in the synthetic textile industries have been studied by a group of experts appointed by the O.E.E.C. Consumption of carbon disulphide, including the amount used in making cellulose film and artificial sponge, amounted to about 170,000 tons in O.E.E.C. countries in 1952. The textile industry consumed nearly 680,000 tons of sulphuric acid (monohydrate) in 1952, most of which was probably used for synthetic textiles.

About 80,000 tons of CS_2 was used in 1952 in making rayon. In the present state of manufacturing techniques, susbstantial recovery of sulphur from this source does not seem likely. On the other hand, staple fibre mills are mostly equipped for recovering the carbon disulphide from the fibre. It may be claimed that, in a staple fibre mill, 40% of the sulphur used is at present recovered as carbon disulphide, and very small quantities of sulphur are recovered as sulphuric acid or sodium sulphide. The O.E.E.C. Working Party considers that it will be possible, in the near future, to raise the proportion of sulphur recoverable in sound economic condition to 50%, compared with a technically possible figure of 65%. The other 35% is lost, either as precipitate on the fibre or in the spinning bath or because it is impossible to seal off the manufacturing equipment hermetically. The quantities involved seem to be too highly dispersed for any recovery process to be economic.

In 1952 staple fibre manufacture accounted for about 115,000 tons of carbon disulphide, about 30,000 tons of which were recovered by the staple fibre mills.

Turning to the question of sulphur obtained from sulphuric acid, the Team states that almost all artificial textile mills using the viscose process possess evaporation plant and most of them are equipped with crystallisation plant. The amount of sulphuric acid saved is already very near the maximum that can be expected from the manufacturing processes used.

Heavy chemical development in Turkey

TURKEY'S nitrogen industry, hitherto neglected, comes in for a good deal of attention in the Turkish Government's 1954 Budget Bill. Installations to be established in Kutahya will use low-grade lignite and will produce over 100,000 tons p.a. of nitrogenous fertilsers and 6,000 tons of nitric acid. A corporation with a capital of T£30 million (T£7.84 = £1) has already been established to meet the cost of the undertaking, which will be T£60 million. The foundations of the factory, in which foreign capital will be involved, will be laid during the current year.

Plans for the manufacture of superphosphates are also advancing rapidly and a new factory will soon start operating in Iskenderun. A foreign firm is participating in the setting up of the factory, which will produce 100,000 to 130,000 tons p.a.

Turkey's cement industry has increased its production by 160% in three years. It has now been decided to expand the Ankara factory and to construct 20 new factories, and the necessary contracts have been awarded; the total construction costs of these factories will amount to T£220 million. It is confidently expected that there will be little difficulty in disposing of the 3 million tons that will be available when the new factories are in operation. Cement imports rose from 150,000 tons in 1950 to 537,000 tons in 1953.

Work is progressing on the Raman oil refinery which will deal with 330,000 tons of crude oil a year; the cost of the refinery is estimated at T£35 million. Another field where considerable expansion is evident is in the production of paper and cardboard from cellulose—from 18,000 tons in 1950 to 50,000 tons in 1953.

Uranium resources of the Commonwealth

AS was pointed out in a brief survey of world uranium production in our August issue, exploitation of uranium ore deposits in certain parts of the British Commonwealth has only recently begun, and these sources of uranium are likely to play a greater part in the future than they have hitherto. A step towards fulfilment of this is the opening, several months ahead of schedule, of Australia's first full-scale plant for the treatment of uranium ores at Rum Jungle in the Northern Territory. The plant has begun large-scale production of uranium oxide which will be sold to the U.K. and U.S., and the output will be about twice as much as originally planned.

Mining operations have been in progress at Rum Jungle since 1953 and there are now large quantities of ore awaiting treatment. And now, just as the new ore treatment plant is put to work, the importance of Australia as a uranium producer is further enhanced by the reported discovery at Pine Creek, about 180 miles south-east of Darwin, of new uranium deposits which are said to be even larger than those now being worked in the Rum Jungle field.

With such promise of abundant uranium in Australia, and the extraction of uranium from gold mine residues going on steadily in South Africa, and further possibilities in other Commonwealth countries, such as Rhodesia, it might be asked whether the Mother Country is also a possible contributor to Commonwealth production of uranium ores. For the answer, we turn to the annual report of the Geological Survey of Great Britain for the year 1953, which has recently been published (H.M.S.O., 3s.), and we learn there that the uranium resources which have as yet been found in

Comical Engineering Terms

A.E.B.

"DUST COLLECTOR"

Britain—almost all in Devon and Cornwall—are not enough to merit the erection of a plant to treat the ore, at a cost maybe of £250,000, but they are nevertheless too large to be considered negligible and efforts towards the discovery of a significant ore-body are going on. Among the old workings explored was the former uranium mine of South Terras; 5,000 ft. of diamond drilling here failed to reveal any payable extension of the ore shoot or the existence of a new one. One new discovery was made, however, towards the end of last year, at Wheal Bray on Bodmin Moor.

One curious fact emerged through the use of a car-mounted Geiger counter and scintillometer equipment which were intended for exploration in the colonies and elsewhere. During early trials of this apparatus in the U.K. a high radioactivity was found along many roads in the Midlands, these roads being metalled by a uranium-bearing slag derived from the smelting of a naturally radioactive phosphate rock to elemental phosphorus. Unfortunately the contained uranium is not recoverable economically, but the slags have proved a cheap and convenient radioactive source for the training of air-raid wardens.

First isopentane plant in Britain

NOW under construction at Stanlow oil refinery, Cheshire, is the first plant to be built in the U.K. for the production of isopentane, an important constituent of aviation gasoline. One of its columns, believed to be the tallest of its kind in the world, will be 200 ft. high. The main contractor carrying out the work for Shell is Head Wrightson Ltd., and the unit is scheduled for completion early in 1955.

Aviation gasoline, which must function efficiently in varying climatic conditions, is a blend of highly specialised components of different characteristics and the isopentane from the new plant will replace similar material which has

hitherto had to be imported for blending up into finished aviation gasoline in the U.K. In this respect the new unit is complementary to the 'platformer' at Stanlow, which came into operation last year, and by means of which certain gasoline fractions are converted into other valuable aviation blending components.

The importance of isopentane as an aviation component lies, amongst other things, in its high octane rating and in its favourable vapour pressure which ensures that the fuel vaporises efficiently in extremes of temperature.

Expected to cost nearly £500,000, the new plant will contribute substantially to the versatility of the refining system at Stanlow, already one of the most comprehensive refining complexes in the world. With an output of nearly 5 million tons a year of various oil products, Stanlow is the largest of the four Shell refineries in the U.K. whose combined annual capacity totals 11 million tons.

In the new unit, debutanised light platformate will be led through a steam preheater to the depentaniser—an 86-ft. column containing conventional bubble-cap trays—where the pentanes will be removed as a top product. The bottom stream—depentanised light platformate—will be run down to aviation component storage. The de-isopentaniser, a 200-ft. column containing 140 Turbo-grid trays, will receive two streams *via* steam heaters, namely the pentane fraction produced above and debutanised straight-run gasoline, and produce isopentane as the overhead stream. The bottom product, consisting of the de-isopentanised straight-run gasoline plus the normal pentanes from the platformate, will be blended into motor spirit. A sphere is being provided for storage of isopentane.

Heat for the process will be supplied by steam—low pressure for preheaters and high pressure for reboilers. The de-isopentaniser operates at 25 p.s.i.g.

Liquid entrainment in distillation

THEORETICALLY the process of distillation involves vaporisation of a liquid followed by removal and subsequent condensation of the vapour formed. In practice this is not always achieved, and the distillate may be contaminated with the original liquid. This contamination has been traced to a variety of causes such as foaming and in particular to entrainment.

The term entrainment has been used to signify the phenomenon whereby a small proportion of the liquor undergoing evaporation is carried over by the vapour as droplets into the condenser, resulting in a loss of the material contained in the evaporator and contamination of the condensate. For example, in concentrating sugar juice, entrainment results in the loss of sugar from the evaporators and in the production of distilled water the prevention of entrainment has been shown to be essential for precise pH work, conductivity experiments, and for water used for surface chemistry.

An investigation into the entrainment of liquid droplets through straight vertical stillheads, during distillation, using fluorescein sodium as an indicating substance, was described by E. Shotton and A. F. S. A. Habeeb in a paper read before the British Pharmaceutical Conference held at Oxford last month. When the Reynolds number for the vapour flowing through the stillhead was below 2,000, indicating streamline or laminar flow, the entrainment was found to be directly proportional to the rate of distillation and to the diameter of the stillhead. As the wall of the stillhead constitutes the main trapping agency it is postulated that the entrainment

will increase as the diameter increases since under these flow conditions the vapour has little radial movement. The entrainment per unit diameter is directly proportional to the Reynolds number in the 30-in. stillheads, but deviations occur in the shorter stillheads of $\frac{5}{8}$-in. and 1-in. diameter, which is probably due to the constriction from 2 in. inducing some turbulence.

As the Reynolds number is increased from 6,000 to 8,000 the entrainment approaches a constant value, irrespective of the diameter, for a given length of stillhead. Under these conditions the flow of the vapour is probably fully turbulent and has a high radial velocity which carries the droplets to the wall. Entrainment has been found to decrease as the length of the stillhead increases from 5 to 15 in. for each diameter, but in the 30-in. stillhead entrainment was highest.

Gross carry-over (creeping film effect) occurred in the 5-in., 10-in. and 15-in. stillheads at an approximately constant value for Reynolds number for each length and was unaffected by the diameter. For the 30-in. stillheads gross carry-over was not achieved for diameters of $1\frac{1}{2}$ in. and 2 in. and in the case of the $\frac{5}{8}$-in. and 1-in. diameter tubes gross carry-over was more nearly related to vapour velocity.

Twenty-one years of polythene

BRITISH output of polythene since its discovery by I.C.I. in 1933 has risen to more than 20,000 tons a year. Much more is made in America and manufacture has been taken up in several other countries.

Behind this commercial progress lies increased knowledge of polythene and its production. In a lecture before the Society of Chemical Industry, Dr. E. Hunter has reviewed present knowledge of polythene and indicated some outstanding problems. He recalled that the early uses required thick sections, and strength—as distinct from flexibility and creep-resistance—was seldom in question. Newer uses, especially film, employ thin layers and strength is important. Increase in molecular weight tends to improve mechanical properties, but increases the viscosity in the molten state essential for fabrication, and compromise has often been necessary. The value of avoiding such limitations will increase as greater use is made of the product's strength. Progress has been made through improved design of fabricating machinery, improved choice of synthesis conditions, and through studies of mixtures of polythenes and other substances.

Efforts to employ lower pressures and temperatures were made more with the object of simplifying manufacture than of improving the product, but no method has appeared for obtaining high polymers of ethylene except at ethylene densities above about 0.35 g./cc. This density can be attained with moderate pressures if low temperatures are used and these conditions, given sufficiently active catalysts, can yield high polymers. Experience has shown that not only high molecular weight but also synthesis temperature is important if the product is to be strong, flexible and extrudable, and no commercial process has been able to avoid high pressures and temperatures for this type of product. Understanding of the reaction has grown steadily with the contemporary growth of the general theory of reaction kinetics of vinyl polymerisation.

Dr. Hunter also pointed out that neither ethylene nor polythene are thermodynamically stable with respect to carbon, hydrogen and methane. In practice, if temperatures are too high, violent exothermic decomposition reactions

intervene, which were a serious problem in the early development. The difficulty of avoiding them is the greater because the polythene reaction is itself strongly exothermic and may be conducted at temperatures which are not greatly below those which induce decomposition. Careful control is now considered very important.

Originally polythene was derived from ethanol, although this appeared unlikely to be the cheapest source for large productions. Since then natural and refinery gases, oil, coke oven gas and acetylene have all been used. At high densities ethylene is relatively incompressible and big pressure increases may be needed to offset the diluent effect of impurities. In addition, some impurities have been found to have specifically undesirable effects. In general, the reaction conditions needed and the type of product obtained have been found to be sensitive to ethylene quality.

Production and uses of selenium

'SELENIUM,' a survey just published (H.M.S.O., 1s. 6d. net), reviews the sources, production and uses of this semi-metal which has become increasingly important to the electrical industry in recent years.

The ability of selenium to rectify alternating current is used in radio communications, broadcasting, domestic radio and television receivers, electrolytic processes and radar equipment and these uses are the predominating ones. Another electrical use is in a variety of photo-electrical cells. It is also used as a decoloriser in glass making and, strangely enough, as a colouring agent in the plastics industry. The expansion of the electronics industry during and since the war has resulted in a shortage of selenium. Any increase in supplies would be welcomed by industry because potential demand for selenium exceeds available resources.

There is no known deposit of selenium as such which is worth mining. The element occurs with sulphide ores and most of it is obtained as a by-product of copper refining. In the electrolytic refining of copper 'anode slime' is formed which contains a fairly high proportion of selenium. The U.S.A. is the biggest producer of selenium, all of it from this process, but its supplies are still not enough for its own industry and it has to import more of it. Most of Great Britain's supplies of selenium come from Canada, again from copper refining plant. There are small quantities of selenium on the market which come from Sweden and Japan, but these are high priced compared with the Canadian selenium.

As noted in these columns in the June issue (p. 162), there is a possible source of selenium in this country which is now being investigated by the Chemical Research Laboratory as a result of a survey of the selenium problem by the Intelligence Division of the Department. Iron sulphide, or pyrites, is used in Great Britain in the manufacture of sulphuric acid. Like copper sulphide it contains selenium. Flash roasting of pyrites is one of the processes which is used to avoid using sulphur as a raw material. The process is fairly new, but its use is expanding and it may produce quantities of selenium which would be worth recovering. The selenium is concentrated in the wastes, dusts and muds from the roasting plant. Little is yet known of the economics of recovery, but waste materials from three plants have been examined at the C.R.L. The materials from one plant contain sufficient selenium to justify the hope that recovery would be worth while.

HIGH VACUUM TECHNIQUE

Vacuum refining; materials of construction; ultra-high vacuum techniques; gauges

By S. L. Martin, M.Sc., F.R.I.C.

(Chemistry Department, University College of the West Indies, Jamaica)

IN keeping with previous practice, no attempt is made to present a full survey of recent literature or to cite examples covering every aspect of the field. Rather, a digest is given of publications which typify current advances and thought, and this is guided by a conscious effort to cover both the fundamental and applied aspects so as to indicate their healthy interplay. Inevitably, there may be claims that better current examples exist—and quite probably many do; the selection is again consciously influenced by the aim of maintaining a common connecting thread as far as is possible.

This review refers to problems of vacuum evaporation of metals (zinc) as an example of industrial applications; to the cosmotron as an example of large-scale vacuum apparatus and inherent problems; to some synthetic polymers of potential use as vacuum 'containers'; to some fundamental advances in 'ultra' high vacuum technique and physical behaviour of certain substances at very low pressures; and, finally, to the ever-present problem of 'gauging' low pressures.

Vacuum-refining: dezincing of lead

Refining of metals by vacuum evaporation or distillation has for some time been practised with the alkali metals, particularly with sodium for use, for example, in sodium vapour lamps, and was developed during the last decade for calcium and magnesium.[1] Of importance are the true vapour pressures of the metal and of contaminants, the influence on the vapour pressures of factors such as surface impurities, alloying constituents or 'bubbling' in the molten state, and possible reactions which may occur with crucible or furnace materials.

Davey[2] has compared some theoretical aspects of the vacuum dezincing of desilverised lead bullion for the batch process (agitated by mechanical stirring) and a continuous process using a turbulently flowing falling film, developed as a full-scale plant in Port Pirie, Australia. After being desilvered with zinc, the lead contains about 0.56% Zn, which is reduced to about 0.05% in the vacuum distillation process. The zinc distillate, to be re-used

for desilvering, may contain up to 10% lead but must be relatively free of oxygen and relatively compact in order not to occupy too great a volume in the vacuum chamber. The evidence is that the zinc atoms diffuse from the quiescent surface through the intervening gas or vapour molecules to the condensing surface; the rate of diffusion of zinc through the molten lead is lower than the rate of evaporation from the surface, necessitating continuous reforming of surface rich in zinc for economical operation. In the batch process this is achieved by mechanical agitation; in the continuous process, by turbulent flow of the molten lead over a heated surface (600°C.) with consequential horizontal evaporation to the condensing surface, as in molecular distillation of oils from a falling film. The condensate may be continuously removed as a liquid (450°C. condenser temperature) or, by rapidly breaking the vacuum for a negligible period, as a solid (about 200°C.). In either process, the zinc diffuses in the vapour state essentially through nitrogen (residual air freed of oxygen by 'gettering action' of initially evaporated zinc) and lead vapour. Optimal conditions are:

(1) Distilland-condenser spacing about 30 cm.
(2) Distilland temperature 600°C. for cold condenser; 30 to 35°C. higher for liquid zinc condenser.
(3) Residual pressure 100 microns or less.
(4) Approximately 0.34 sq. ft. evaporating surface for each ton per hour bullion to be dezinced from 0.56 to 0.05% Zn at 600°C. and 100 microns with a cold condenser.

Using published vapour pressure and free energy data, Davey developed theoretical expressions for per cent. zinc left in lead as a function of time for different degrees of vacuum, distilland and condenser temperatures—both for batch and for falling film processes where the effect of flow rate was also studied. In the batch process at 600°C. and 100 microns with 30-cm. path the theoretical time to reduce from 0.56 to 0.05% Zn is 1,460 sec., whereas the actual process time is 5 hr., or 12 times as long. This is felt to be due

to precondensation of zinc in the vapour space, the zinc falling back on to the distilland because of the upright distillation path. The agreement between theory and practice in the falling film process is fairly good because, although precondensation still occurs, the resulting zinc instead of falling back on to the distilland falls parabolically on to the distillate, since the distillation path is horizontal. There is evidence that this precondensation effect results not primarily from a fall in temperature consequent to adiabatic diffusion, but rather to the condensation of zinc vapour on liquid globules of lead present in the free space. This postulate appears reasonable, since the streaming zinc vapour would act as a pump (compare the mercury diffusion pump) and drag lead atoms into the stream where they may 'cluster' and form liquid globules.

Davey has further generalised his description of the process to apply to any similar vacuum distillation process at 10 to 100 microns where the distilling element (zinc) has a partial pressure of 100 microns or greater and the non-volatile constituents (lead) one of less than 10 microns, and where the evaporating and condensing surfaces are parallel and there is unrestricted vapour flow of fairly short path.

Vapour pressure and free energy data for metals

As indicated above, knowledge of vapour pressure data is of importance in vacuum metallurgy. Compilations of such data are available,[5, 6, 7] but should be used with care, since the actual values of vapour pressures may depend strongly on purity and pre-history of the metal. A point that is often overlooked also is that the vapour pressure of a metal over an alloy depends on the activity coefficient of the metal in the particular alloy. Activity coefficients for alloy systems are usually calculated from e.m.f. measurements. It has recently been shown that vapour pressure data calculated with these coefficients agree very well with experimentally determined values for several alloy systems.[3, 8] For example, McCabe *et al.*[3] quote results which indicate the following approximate data for silver in silver-gold alloys at 1,000°C.:

At. % Ag	Estimated acty. coeff. of Ag	Approx. V.P. mm. Hg × 10⁴
100	1.0	52
77.5	0.95	40
62.0	0.8	22
38.8	0.55	12
19.5	0.45	4.5

These results suffice to show the likely importance of such corrections. (It should be noted that for zinc in lead Davey used vapour-pressure data for pure metal.)

In many instances, particularly where the metal being distilled is formed *in situ* by a chemical reaction, free energy data are of more use than exact vapour pressure data, and are essential for estimating equilibrium vapour pressures of desired metal (Ba) over reaction mixtures of the type:

$$3 \text{ BaO (s)} + 2 \text{ Al (l)} \rightleftharpoons 3 \text{ Ba (vap.)} + \text{Al}_2\text{O}_3$$
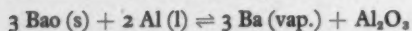
in order to evaluate commercial potentialities of such production methods. Besides the free energies of formation (for instance, of the oxides above), free energies of vaporisation of the metals as functions of temperature are required. Evans[4] has recently used Brewer's data[7] on free energy functions for solid, liquid and gaseous elements and heats of vaporisation to calculate standard Gibb's free energies of vaporisation (from the liquid) and sublimation (from the solid) as functions of temperature; his data are shown in Fig. 1; Ti, W, Mo and Pt were excluded because of uncertainties in the high heats of vaporisation data, Co, V and Zr because of general uncertainty, and As, Sb and Bi because of the polyatomic nature of their vapours.

Evans estimates for the example cited above a Ba vapour pressure of about 27 mm. Hg at 1,500°K. He further points out that the data may be used cautiously to estimate orders of vapour pressures of the metals by means of the approximate Clausius-Clapeyron equation:

$$\log_{10} p \text{ (mm. Hg)} = \frac{-\triangle \text{F}^{\circ}{}_T}{4.57T} + 2.88$$

Rossman and Yarwood[9] have further emphasised and illustrated the difficulties of assessing true evaporation rates from molten liquids *in vacuo*. They were primarily concerned with experimental requisites to establish basic data rather than with industrial processes. The essential difficulties (applicable also industrially) concern establishment of constancy and precise evaluation with respect to:

(1) True evaporating surface (affected by bubbling and 'spread' on crucible surface).

(2) Temperature of evaporating surface.

(3) Condensation coefficients—fractions of number of atoms condensing to the total number striking the surface per unit time.

(4) Crucible material and shape.

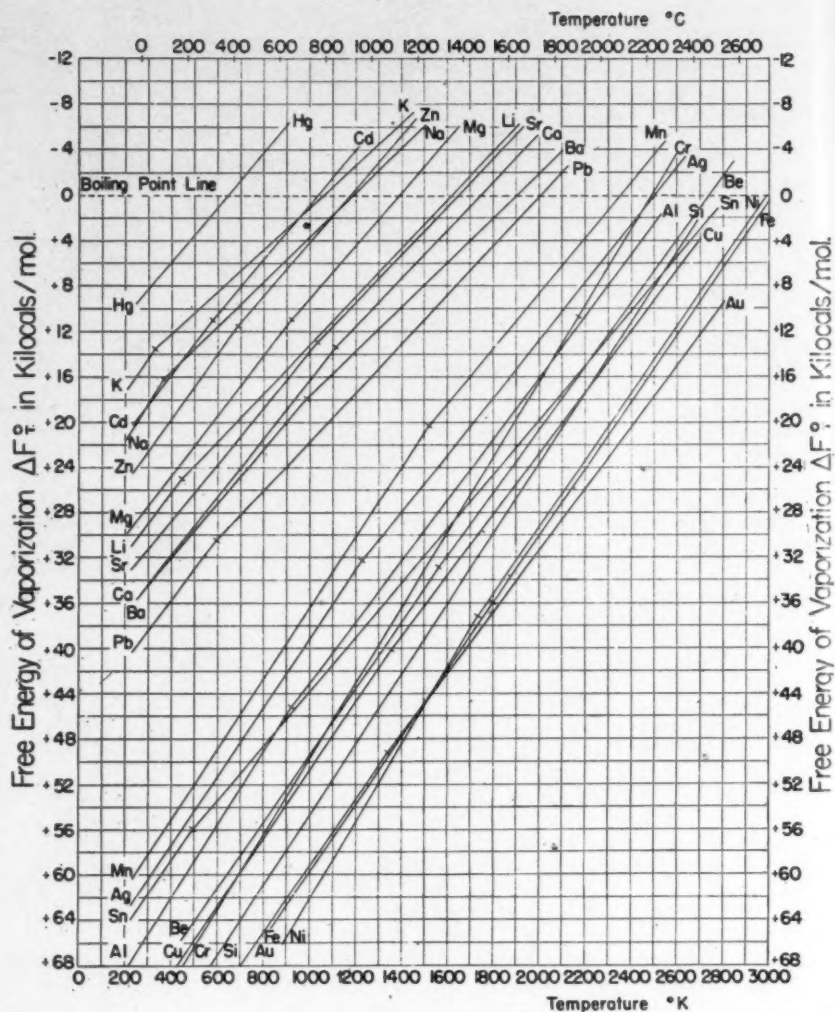They describe a carbon crucible and furnace arrangement relatively free from

Temperature °C

Free Energy of Vaporization ΔF°T in Kilocals/mol.

Free Energy of Vaporization ΔF°T in Kilocals/mol.

Temperature °K

Courtesy: Journal of Metals

Fig. 1. Free energies of vaporisation of solid and liquid elements ($\triangle$F°T). Break in line occurs at melting point (Evans[4]).

objections (Fig. 2), permitting optical observation of temperatures through a hole in the nickel shield and resisting attack or wetting by Au, Ag and Hg but not Al. They arrive at an expression, developed by analogy with radiation problems, allowing for corrections due to the space in the crucible above the evaporating surface (shape factor). Even with mercury, however, the results were inconsistent, indicating a 'best value' of the accommodation coefficient of 0.7 (*cf.* more accepted value of nearly unity). With silver and gold the scatter of the results of 47 experiments did not warrant estimates (spread at 1,200 to 1,300° was 100- and 10-fold, respectively, for 29 experiments). It was felt that the inconsistency was due to surface contamination with carbon particles and inaccuracies (5°) in temperature measurements. Condensation coefficient values (*a*) indicated for silver were as low as 0.1, which emphasises their probable importance in estimating rates of evaporation *m* (g./sq.cm./sec.) from saturated

vapour pressures (*p*) from the generally accepted equation:

$$m = a \, p \, (M/2\pi RT)^{\frac{1}{2}}$$

where *M* is the molecular weight and *R* the gas constant in units appropriate to *p*.

Materials of construction

An example of difficulties with constructional materials with large-scale equipment, as distinct from the experimental apparatus above, has recently been given in some detail. This concerns the design of vacuum chamber[10] and pumping system[11] for the 2- to 3-billion electron-volt proton synchrotron (*Cosmotron*) for studies on fundamental nuclear particles at Brookhaven, U.S.A. The problem was to provide a rectangular-sectioned (approximately 7 × 32 in.) vacuum chamber of non-ferro-magnetic material positioned circumferentially in an orbit of radius 30 ft., and capable of being maintained at a maximum pressure of 5 × 10⁻⁶ mm. Hg despite the requisitely thin walls. Ceramics
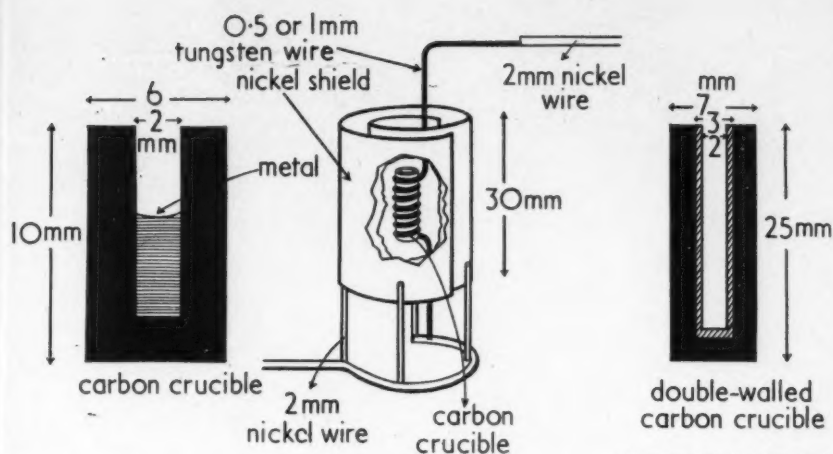
Fig. 2. Double-walled carbon crucible with heater and radiation shield, for experimental work on rates of evaporation of metals.[9]

Courtesy: Brit. J. App. Physics

proved unable to withstand the pressure differential, and plastics had too high an outgassing rate. A continuous metal surface was undesirable because of the need to break up eddy currents. The final design used stainless-steel end plates with stainless-steel bars 2-in. wide spanning the 32-in. gap (608 of them to a quadrant), a newly developed low-vapour-pressure rubber—*Myvaseal*—providing an airtight cover. The volume of 300 cu. ft. is evacuated by twelve 20-in. oil-diffusion pumps backed by 8-in. booster pumps and rotary pumps. The estimated areas of exposed *Myvaseal* are 44 sq. ft. for gaskets and 68 sq. ft. for the membrane (reduced to this from a total of 1,100 sq.ft. by greasing the steel bars). Over a period of

15 months' operation at about 65 hr./week no serious leaks developed.

It is interesting to note that some tests with thin *Inconel* strips stitch-welded to large membranes were encouraging. The poly-trifluorochloroethylene plastic *Kel-F* grade 300 (M. W. Kellogg Co.) was found to have low vapour pressure and outgassing rate, and to be particularly suited for moulding small washers and insulators to be used inside the chamber, but had insufficient elasticity for use as a membrane. *Myvaseal* was nearly as good as *Kel-F* in the former respects, and much better in the latter. Both were better than latex or *Neoprene* rubbers. *Myvaseal*, though somewhat semiplastic in nature, has advantages over *Neoprene* as a gasket material and can

be vulcanised at 165°C. with a *Myvaseal* cement (Distillation Products Inc.).

Duncan and Warren[12] have described some preliminary work with another polymer of potential use as a constructional material where special factors such as water vapour adsorption or attack by substances under investigation preclude the use of glass or metal. Polythene is available in powder form, sheet, tubing and as a grease from I.C.I. Ltd. It melts at about 110°C., so can only be used at low temperatures. It is easily joined to itself and to glass, ceramic and metal by welding in a hot nitrogen jet, and other materials can be readily coated with it by being plunged hot (200°C.) into the powder and then heated at 120°C. Complete evacuation is withstood with $\frac{1}{8}$-in. thickness at up to $\frac{1}{2}$-in. diam., and $\frac{1}{2}$-in. thickness at 6-in. diam. Polythene is slightly permeable to gases and water vapour, but with suitable thicknesses and clean surfaces pressures of 10^{-6} mm. Hg are attainable with an overnight rise of not more than 10^{-4} mm. Hg; moreover, on opening to air, repumping for about $\frac{1}{2}$ hr. suffices to remove adsorbed gases, compared with some hours for glass and metals. It is inadvisable to immerse parts in liquid air. The grease is unsuitable for use at pressures below 10^{-3} mm. Hg. Vessels, valves, taps and even manometers have been constructed. Membrane, 0.02-in. thick, used as a diaphragm, withstands pressure differences of 100 mm. Hg and is sensitive to pressures around 10^{-3} mm. Hg. Figs. 3 and 4 illustrate the construction of vessels and valves.

On the other hand, Ennos[13] reports some results not favourable to the use of polythene in demountable kinetic vacuum systems where contamination of specimen surfaces is important (as in electron microscopes). He tested a variety of organic materials in a system where a stainless-steel target was bombarded with electrons and the contaminating power assessed by the thickness of the resulting carbon deposit. Table 1 shows some of his data (rubbers were all boiled in aqueous and alcoholic potash, polythene washed in detergent, plastic sheets cleaned, brass cleaned in acid, and aluminium in potash solution).

The authors show that contamination is considerably reduced with a coolant at −15°C. before the specimen chamber, or by keeping the specimen at 200°C., and consider the complete elimination of grease and rubber gaskets desirable.

Ultra-high vacuum techniques

It is obvious that permissible contamination from materials of construction depends on the particular application. It is a natural next step to consider what is the best degree of vacuum at present obtainable, assuming complete absence of contaminating material. In the electronic-tube industry the most stringent demands for very high degrees of vacuum are encountered, and it is therefore natural to expect advances in this respect to originate in the

Courtesy: Brit. J. App. Physic
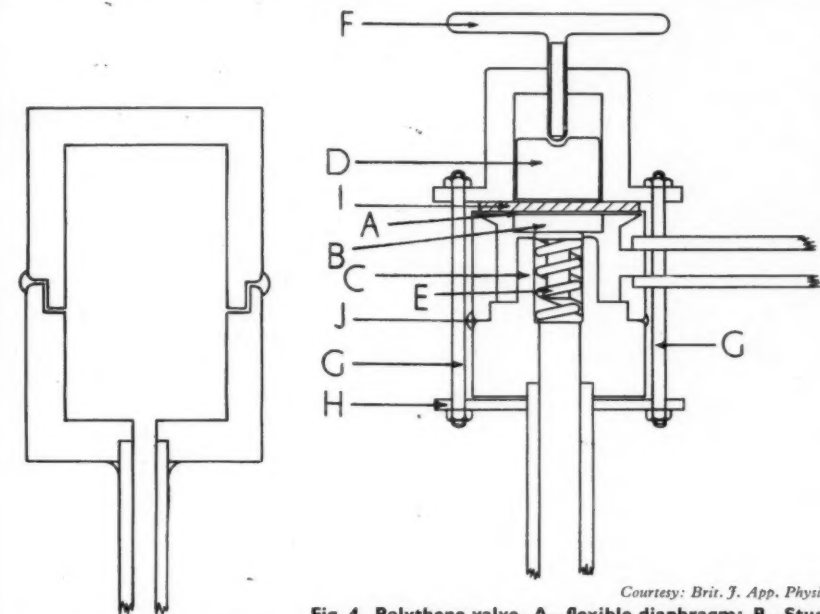
Fig. 3. Construction of small polythene vessels, by use of lapped joint and seal.

Courtesy: Brit. J. App. Physics

Fig. 4. Polythene valve. A—flexible diaphragm; B—Stud; C—valve seating; D—brass plunger; E—polythene-coated metal spring; F—brass key; G—brass clamping rods; H—brass retaining ring; I—neoprene washer to protect polythene from abrasion by plunger; J—welding junction.

research laboratories of such concerns. Basic to this demand is a satisfactory method of measuring very low pressures. Claims that pressures as low as 10^{-9} mm. Hg could be measured with ionisation gauges have been received with some scepticism by various authors outside the U.S., amongst whom has been the present author in previous reviews of this series, because of the known vagaries of ionisation gauges. However, Alpert[14, 15] has recently described a new set of ultra-high vacuum techniques evolved in the Westinghouse Research Laboratories, and referred to unpublished work of Nottingham along similar lines elsewhere. The evidence is now strong enough to convince previous doubters that with these specialised techniques and gauges indications of pressures less than 10^{-12} mm. Hg can be obtained, and that one of the limiting factors in the attainment of very low pressures is the diffusion of helium from the air through the glass systems.[15]
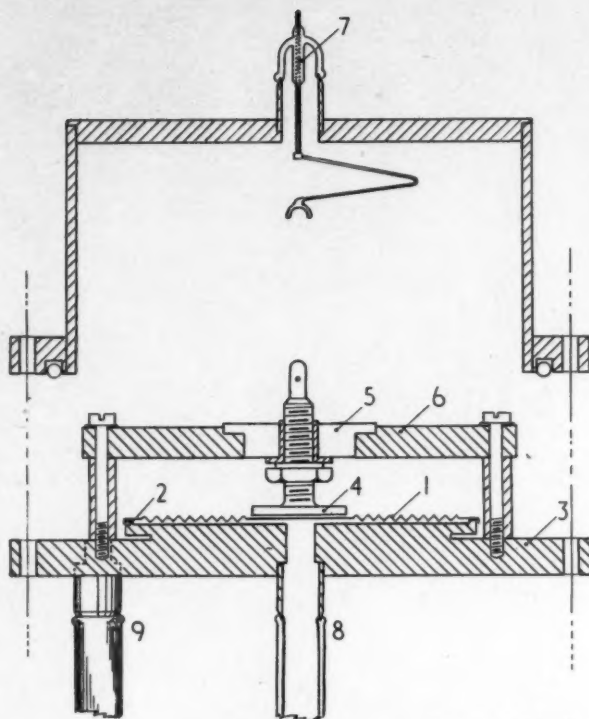
The essential points described by Alpert[14] for achieving working pressures between 10^{-9} and 10^{-10} mm. Hg 'in a manner which is in no case more difficult than the procedures conventionally used to attain the usual " good vacuum " of 10^{-7} mm. Hg,' and this without the use of getters, traps or refrigerants, are:

(1) Use of a sensitive gauge—the Bayard-Alpert ionisation gauge designed to eliminate the ' residual ' effect of soft x-rays.

(2) Elimination of grease and pump-oil vapours without traps, by using a special vacuum valve, capable of high-temperature bake-out; in this a highly polished Kovar nose is forced under 1 to 2 tons' thrust into an orifice in a copper cup by a special screw-thread arrangement. Leak rates of 10^{-10} litres/sec. or less can be maintained for a year. Kovar-to-glass seals are used.

(3) Use of unitised base pads for assembling the system so that the whole of it can be baked out (overnight) by specially designed unitised furnaces at 400 to 500°C. after being evacuated with a glass fractionating oil-diffusion pump. The ionisation gauge is then degassed, and the valve closed to seal the system from the pumps.

(4) Final evacuation of the isolated

Table I. Thickness of Contamination in Å Units on Bombarded Steel under Standard Conditions for Various Materials (Ennos[13]).

Material	Å	Material	Å
Apiezon B oil	1,700	Polythene	1,300
Silicone DC703 oil	500	Perspex sheet	< 50
Apiezon M grease	1,500	Bakelite sheet	< 50
Apiezon W wax (cold)	< 50	Brass strip	700
Natural rubber	1,100	Brass strip (cleaned)	< 50
Rubber gasket cords	700	Aluminium strip	700
Black Neoprene (heavily loaded)	< 50	Aluminium strip (cleaned)	< 50
Edwards' O-rings	600		

system by the pumping action of the gauge itself. It has been found that the well-known ' electrical discharge gettering ' action, due to the trapping of positive ions of gases (formed by electron impact) in the negatively-charged surfaces, and the ' chemical ' clean-up action resulting from interaction of gases with the hot cathode, can rapidly reduce a well-outgassed system to very low pressures and maintain them for very long periods (75 days at 10^{-9} mm. Hg in one case without evidence of saturation).

Even with the most careful precautions it was not possible to achieve total pressure indications below 4×10^{-11} mm. Hg, even though there was no indication of any adsorbable gas. This lead to a search for a source of some non-adsorbable gas.[15] It was found that evaporation of tungsten from the cathode at 2,300°K. set a low limit of 10^{-12} mm. Hg (this work indicates a use for such systems to measure low vapour pressures). With a specially developed simple version of an ' omegatron ' mass spectrometer gauge for measuring partial pressures of residual gases in terms of their mass units, it was shown that the gas in this case was helium. The rate of diffusion of helium through glass was next measured; this, coupled with the calculable rate of helium clean-up by the ionisation gauge, accounted for the measured equilibrium or ultimate pressure of 4×10^{-11} mm. Hg.

It must thus be concluded that lower total pressures will only be attained if the rate of diffusion of helium into the system

Fig. 5. Section of diaphragm capacity gauge-head (Cooke and Danby[19]).

can be reduced, or its rate of clean-up by the gauge increased.

Gauges

Alpert[14] summarises the necessary technique with the Bayard-Alpert ionisation gauge, now available commercially, and describes a suitable electronic circuit for it. The novel features of the gauge are two filaments external to the grid, and a thin wire ion-collector within the grid intercepting only a small fraction of the soft x-rays from the grid. One filament is used for the ' flash filament ' technique of Apker to gauge the presence of gases adsorbable on the tungsten filament. The gauge can be calibrated directly by expansion methods in conjunction with a diaphragm gauge previously described;[16] this uses a 0.005-in. Kovar sheet (separating the ultra-high vacuum side from another system including an ordinary manometer), the electrical capacity between which and an electrical probe affords a null-reading manometer accurate to 10^{-2} mm. Hg over the range 0.1 mm. Hg and upwards.

The simple omegatron referred to above consists[15] of a 1-cc. box with two r.f. faces (corresponding to the dees of a cyclotron), into which is shot a narrow beam of ionising electrons parallel to the direction of a magnetic field. The ions are accelerated in orbits of increasing size at impressed r.f. frequencies inversely proportional to their masses, and eventually hit a small ion collector giving an ion current the relative intensity of which reflects the proportion of the particular ion present; the mass

number range of 40 to 1 can be covered with r.f. frequencies 50 to 900 kc/s.

Such gauges are of specialised rather than general use. Bradley[17] has suggested the use of commercially available thermistor beads for sealing in the top of the capillary of McLeod gauges—instead of the more common micro-Pirani attachment —to increase the sensitivity in the lower ranges. The technique is said to be simpler, normal Wheatstone bridge resistance circuits can be used, and the reproducibility over the range 1 to 10^{-7} mm. Hg is good.

The diaphragm electrical capacity type of gauge has been receiving some attention because of robustness and adaptability to the sealing-off of measurement parts of the system from gauge fluids. Winfield[18] used an Ag-coated diaphragm, movable (by alternating gas pressure) in relation to a fixed brass disc, for continuous measurements on rates of adsorption of vapours by solids. The increase in capacitance when the diaphragm moves towards the disc was made to alter the frequency of a tuned oscillator, and the frequency change was converted to a voltage change (by a discriminator) which was amplified and fed on to an oscilloscope tube; the pressure range can be altered by changing the tuning and amplification factor, to cover the range 0.1 to 20 mm. Hg in 2-mm. stages. Cooke and Danby[19] have described a similar type of gauge covering the range 1 to 100 micron with an accuracy better than 0.5 micron, the head of which is shown in Fig. 5. A Cu or Ag corrugated diaphragm about 0.001-in. thick (1) is soft-soldered (2) just above the lead-in tube (8) from the vacuum system, and about 0.01 in. from the upper condenser plate (4) mounted on (6) via insulation (5). When the cover is put on, contact is made by the top lead passing out through insulation (7), and the external space is kept evacuated through tube (9). The capacity change is converted with a simple one-valve circuit to a frequency change of an electron-coupled oscillator; each pressure is then associated with a definite frequency measurable by bringing a tuned circuit including a precision variable condenser into resonance with it, and the condenser scale can be calibrated in terms of pressure.

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EVAPORATION

Caustic soda; cane sugar; scaling of surfaces; concentration of heat-sensitive materials; submerged combustion; separator design; heat transfer coefficients

By J. M. Coulson, M.A., Ph.D., A.M.I.Chem.E.
and M. J. McNelly, B.Sc.(Eng.), A.C.G.I.

DURING the past year there have been a number of papers concerning the flow mechanism in evaporator tubes and boiling film heat transfer coefficients. However, the entire solution of the problem of flow in an evaporator tube is far from complete. The scaling of heating surfaces especially in the sugar industry has again received considerable attention and the problem of evaporation of heat-sensitive liquors continues to be a major interest.

Industrial plant operation

(a) **Caustic Soda.** A plant for the production of anhydrous caustic soda at Dow Chemical Co., Pittsburg, which has completed nearly four years commercial operation using a continuous finishing process, is described by W. Badger and F. Stanford.[1]

The process is carried out in a climbing film evaporator constructed of nickel and heated by 100 p.s.i. *Dowtherm* vapour. This method supersedes the batch finishing process using direct-fired cast iron pots.

A continuous feed of 50% NaOH to the unit can be concentrated to greater than 99.5% solids in a single pass. Shading of the product (*e.g.* removal of the metallic impurities) is found to be unnecessary so long as the chlorate concentration is kept very low by previous reduction of any chlorates present in the feed by a compound such as glucose. This chlorate otherwise yields nascent oxygen on thermal decomposition and this readily oxidises the nickel of the plant.

The advantages of this type of plant are stated to be: only one-fifth of the capital cost, double the fuel economy, lower maintainance and replacement costs, and increased suitability for automatic control as it is a continuous process. There are now five such units in operation of which the first two are completely jacketed with 100 p.s.i. *Dowtherm* vapour. In the more recent units this jacketing has been replaced by 3 to 4 in. of lagging as the vapour leaving the evaporator tubes has been found to possess sufficient super-heat to maintain the plant above its freezing point. This eliminates the need for pressure vessel type construction of the separator etc. for withstanding 100 p.s.i. *Dowtherm* and is consequently far more economical.

(b) **Cane Sugar.** Suitable operating conditions for the first vessel in a cane juice evaporation plant are described by C. Perk.[2] The need for a minimum retention time for the cane juice in the first evaporator vessel as a result of its relatively high temperature is stressed and consequent advantageous designs are discussed.

The Kestner type long tube evaporator with relatively small diameter tubes is ideal owing to the large ratio of heating area to volumetric hold-up capacity. Mention is made of the impossibility of unlimited concentration by increased steam temperature as the liquor level gradually sinks to a minimum value corresponding to a maximum ratio of evaporation rate to feed rate. This assumes a constant feed liquor head. However, free recirculation is possible and this will enable higher degrees of concentration to be reached. Mention is also made in the article of vapour bleeding for feed preheating.

Scaling of heating surfaces

N. Schmidt and L. Wiggins[3] describe work carried out to determine the efficiency of a sodium salt of ethylenediaminetetraacetic acid for the removal of evaporator tube scale deposited from sugar cane juice. In the sugar industry, scale deposition is very rapid, *e.g.* 1 mm./week and can cause a major hold-up in the middle of the cane processing season. This salt is a so-called ' chelating agent ' and is used in analytical

chemistry for the determination of the hardness of water as it forms stable water-soluble complexes with metal ions such as calcium and magnesium. It is salts of these metals, which form the scale in sugar cane juice evaporators and therefore a technical grade of this salt, with the trade name of *Versene*, has been put on the market.

Versene was found to be very effective for dissolving the scale and theoretically 1 lb. of the salt should combine with 0.172 lb. calcium oxide or 0.416 lb. calcium sulphate. However, from economical considerations, it is over twice as expensive as the usually employed methods of mechanical and chemical cleaning. There are however, some indirect benefits, such as the condition of the tube surface which was considered to be less likely to encourage further scale deposition than an acid- or mechanically-cleaned surface. Also *Versene* cuts cleaning time.

The reduction in corrosion and scaling attainable by evaporation at very low pressures is emphasised by Kelly,[4] who states the case of corrosion of a mild steel plant which is reduced from 0.045 to 0.01 cm./year, by reducing the evaporation temperature from 60°C. to 20°C. for a salt solution.

Kleinert, *et al.*,[5] describe scale formation in the viscose fibre industry where the deposition of a mixed salt of sodium and calcium sulphate is the main cause of scaling. The solubility of the salt and methods of prevention of its deposition are given.

The following methods of combating scale-forming liquors in evaporation problems are listed by Lindsey,[6] in an article on equipment available for carrying out evaporation, *e.g.*:

(1) For calcium sulphate which has an inverted solubility curve, forced precipitation of the salt by preheating the feed to well above the evaporation temperature by live steam injection.

(2) For sea-water scale the addition of a compound causing a fluffy scale deposit which is removable by cold shocking.

(3) Another method of scale removal is to construct the heating surface so that it can be flexed by changing the internal pressure, thus causing the scale to drop off.

A further method of combating scale-forming liquors is to entirely eliminate the heating surface by the use of a direct-heated type of evaporator. B. N. Reavell[7] refers to the use of spray driers (Fig. 1) and submerged combustion units in connection with this subject.

Concentration of heat-sensitive materials

A large proportion of the liquids concentrated in industry by evaporation in bulk are heat-sensitive, and B. N. Reavell[8] outlines some of the units available for the process. As noted in the article the term heat-sensitive is a rather vague one, but in general substances termed as such are very dependant on a time factor as well as the
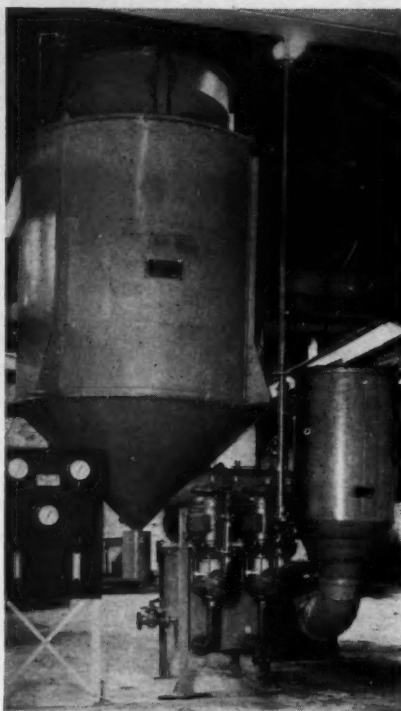
Kestner Evaporator & Engineering Co. Ltd.

Fig. 1. A typical spray evaporator
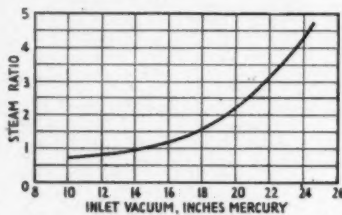
Hick Hargreaves & Co. Ltd.

Fig. 2. Thermo-compressor performance curve with varying inlet vacua giving lb. of steam at 130 p.s.i.g. per lb. of vapour compressed to 1 p.s.i.g.

temperature level. This is well demonstrated by the fact that a golden-coloured syrup was first prepared by the sugar industry in a climbing film evaporator in place of the darker syrup produced in the equipment used up to that time. The reason for this was the much shorter reten-

Table I

Type	Approx. installed cost	Cost steam per annum	Net saving per annum compared with single effect
	£	£	£
Single effect	5,000	40,300	—
Double effect	7,000	21,400	18,900
Double effect with vapour recompression	9,000	13,700	26,600
Triple effect	10,000	14,300	26,000

Source: Reavell.[7] *Courtesy: Kestner Evaporator and Engineering Co. Ltd.*

tion time in the heating zone of a climbing film evaporator where the colour degradation occurs.

It is pointed out that by using a double effect evaporator with vapour recompression a lower maximum temperature is possible than would be the case in the first effect of a triple effect system there is also the advantage of higher thermal efficiencies obtainable with this type of unit. Furthermore the lower capital cost is in favour of this unit and Table 1 shows its overall economy for the proposed duty of concentrating 6 tons/hr. feed containing 10% solids to 50% solids for a 160 hr. week.

The net saving excludes a plant depreciation of approximately 15% of the capital cost but this will make little difference. An indication of the steam usage of a thermo recompression unit is given in Fig. 2.

The heat pump evaporator (Fig. 3) has some similarities to a vapour recompression unit and a similar heat economy. In this unit the same working fluid transfers heat both to the boiling liquid and from the condensing vapour. Consequently, whatever the temperature level at which evaporation is carried out adequate condensation may be obtained without separate refrigeration for cooling the condenser. This would normally be required when concentrating or distilling such liquids as acetone or amyl acetate.

Equipment

No basically new types of equipment have appeared during the past 12 months, but considerable operating experience has been obtained on some of the more recent types.

E. Lindsey[6] describes the various types of evaporators available and indicates their respective performances, with the aim of giving guidance for the solving of evaporator problems. Three of the principal types, namely, climbing film, falling film, and forced circulation units are compared. The simplicity and economy of submerged combustion is emphasised and two of the newer evaporator units critically discussed.
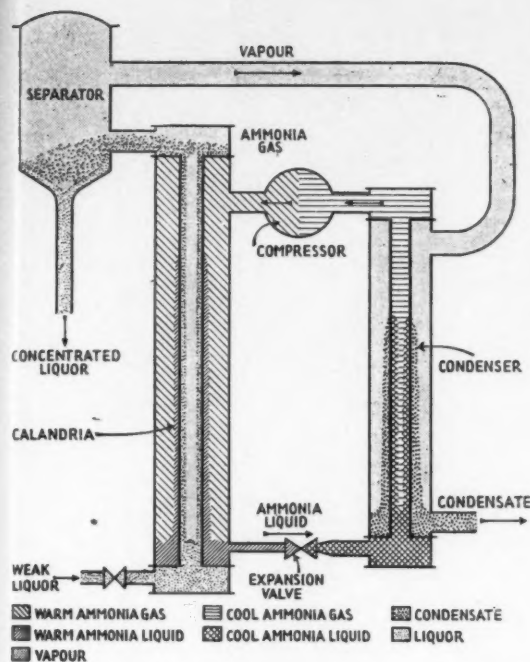
These new units are the Turba-film and the Rosenblad switching evaporator as described in the review in this journal last year. The former is a large diameter falling film unit with an internal agitator. This agitator makes the unit complex and owing to the narrow clearances required, also very expensive. The units so far available are of relatively small capacity and their application seems to be for small quantities of very viscous (up to 100,000 c.p. at the evaporation temperature) and heat-sensitive materials. The latter unit, as its name implies, regularly reverses the evaporating and steam-heated surfaces in order to reduce scaling by dissolution with the steam condensate during alternate cycles. This novel unit is very expensive as not only does it consist of a duplicated set of equipment but also complicated switching gear for flow reversal at the end of each cycle.

Finally, various types of energy saving

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Fig. 3. Diagram of a heat pump evaporator.

heat pump cycles are described and also the design of separators is considered.

Kelly[4] states that the present tendency in forced circulation evaporator design is to increase the circulation velocity so reducing the surface area required. However, it is possible to obtain heat transfer coefficients approaching these values by the use of falling film units.

Submerged combustion

The use of submerged combustion evaporators (Fig. 4) is an effective method of concentrating strongly corrosive or scaling liquors as stated by B. N. Reavell.[7] In his paper attention is also drawn to the fact that the boiling point may be reduced by a partial pressure effect in this type of equipment as is illustrated by the following figures:

For a total pressure of 760 mm. Hg water boils at:

84°F. using 100 B.Th.U./cu.ft. blast furnace gas
86°F. using 140 B.Th.U./cu.ft. blast furnace gas
90 to 92°F. using 500 B.Th.U./cu.ft. coal gas

Separator design

There continues to be a lack of data on the performance and design of vapour-liquid separators suitable for use with evaporation equipment, where a certain amount of entrainment is always present. The more stringent application of the anti-pollution laws and the special case of radio-active liquors has aroused great interest on separator design. The cyclone separator appears to be the most effective type, and B. N. Reavell[7] describes a test of one used in connection with a radioactive liquid which gave an entrainment loss of less than 50 p.p.m. when working at the full rated capacity of 1,200 lb./hr.

E. Lindsey[6] gives a number of diagrams of the types of separators in use in connection with evaporators but no very definite indication of their performances.

Heat transfer coefficients

(a) Plant data. The literature contains a very large amount of data on the heat
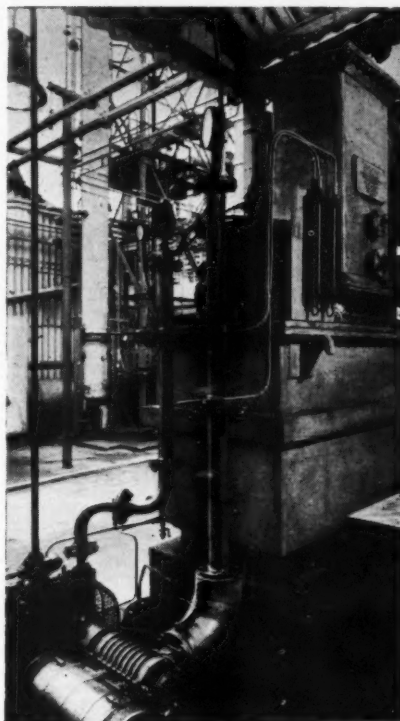
May & Baker Ltd. and Nordac Ltd.

Fig. 4. A 'Swindin' submerged combustion evaporator. This unit is for concentrating spent sulphuric acid containing hydrochloric acid.

transfer coefficients measured between boiling liquids and various heating surfaces. However, until a thoroughly satisfactory method of predicting the coefficients is found, estimation of the values likely to be obtained, by comparison with practical data obtained under similar conditions, is the only possibility. In this case the more data available the more reliable is the method, especially with the large range of conditions that are possible.

E. Kirschbaum[8] investigated the heat transfer coefficients for sugar solutions and water boiling in a vertical tube 4 m. long and 4 cm. diameter. Tube wall thermocouples were used in order to determine the mean wall temperature, and a travelling thermocouple to determine the non-boiling length and the mean boiling temperature.

Liquid and wall temperature distribution curves are plotted against tube length and show the non-boiling length to vary between 25 and 65 % of the total tube length. The mean film heat transfer coefficients are plotted against temperature difference for various pressures and feed rates and an empirical correlation is given for the effect of temperature difference on each of the solutions.

E. Piret and H. Isbin[9] give heat transfer data for six liquids boiling in a vertical natural-circulation evaporator at atmospheric pressure. The evaporator tube is 1.068 in. internal diameter and 4 ft. 10 in. long, and externally wound with three electric heaters. Six tube wall thermocouples are used in order to determine a mean wall temperature for calculating the film temperature difference. The recirculation rates for this particular configuration were measured using a flow meter with a negligible pressure drop.

An indication of the effect of recirculation rate on the heat flux has been given, but the temperature difference does not seem to have been kept constant, so the true effect of circulation rate remains unknown. Also, a correlation has been shown to cover 91.3% of the results within 10% but it is stated to be an empirical relation of limited application.

J. Myers and D. Katz[10] present a number of useful sets of data for boiling heat transfer to various refrigerants boiling outside ¾-in. diameter plain and finned tubes, at a series of pressures. They also produce a dimensionless correlation for some of the results, stating that these liquids must have similar interfacial relationships to the heating surface whereas the others presumably have not.

(b) Correlation of plant data. Several correlations of practical data have been published in the last few years, but the problem of the effect of heating surface conditions has been a major difficulty and has tended to limit their applicability.

A dimensionless equation correlating the film heat transfer coefficients to nucleate or submerged boiling liquids has been derived by M. McNelly,[11] the general method used being that of dimensional analysis.

The equation:

$$\frac{hd}{K} = 0.225 \left(\frac{c\mu}{K}\right)^{0.69} \left(\frac{Hd}{L\mu}\right)^{0.69}$$
$$\left(\frac{Pd}{\sigma}\right)^{0.31} \left(\frac{\rho L}{\rho V} - 1\right)^{0.33}$$

$h =$ the heat transfer coefficient.
$d =$ some linear dimension (of which h is not a function).
$K =$ the liquid thermal conductivity.
$c =$ the liquid specific heat.
$\mu =$ the liquid viscosity.
$H =$ the heat flux per unit area of surface.
$L =$ the latent heat of vaporisation.
$P =$ the vapour pressure of the liquid at its boiling point.
$\sigma =$ the vapour-liquid interfacial tension.
$\rho L =$ the liquid density.
$\rho V =$ the vapour density.

(N.B.—All physical properties should be for the substance at its boiling point).

is shown to correlate the data of some 10 different groups of workers, covering 14 different liquids, whose results are published in the literature. The range covered extends from liquid nitrogen boiling at atmospheric pressure, to benzene boiling at conditions very near to its critical point; and from refrigerants boiling just above atmospheric conditions to water boiling at considerably reduced pressures. The accuracy of the correlation is stated to be comparable with that of the data used, *e.g.* mean error 20% of the heat flux. This range covers the effect of a very varied range of heating surfaces used by the authors of the data. Furthermore, the effect of this variation on the overall coefficients which are the ones which determine a plant's performance will be greatly reduced and should therefore, come well within design tolerances.

(c) **Flow mechanism in evaporator tubes.** B. Harvey and A. Foust[12] have carried out a mathematical analysis of two-phase flow such as may occur in an evaporator tube. Their intention was to predict the temperature variation along the length of a long tube evaporator, which they operated to check their predictions for water boiling at various pressures. The evaporator used contained seven $1\frac{1}{2}$-in. diameter tubes, 20 ft. long, and the following assumptions were made in order to carry out the analysis:

The vapour and liquid travel up the tube as a homogeneous mixture.

There is no slip between the vapour and the liquid.

The heat is transferred uniformly along the length of the evaporator tube to the boiling liquid.

This analysis predicts a critical flow condition but as was pointed out by M. Coston (Swenson Evaporator Co., Philadelphia, Pa.) in the discussion following the paper, the conditions considered in this paper are well above those for all but one or two

exceptional evaporators that are operating at the present time. However, the temperature variations below this critical range are stated to be predictable to within an average error of 4%, using this rather complex mathematical solution for the one dimensional flow equations for flow in an evaporator tube.
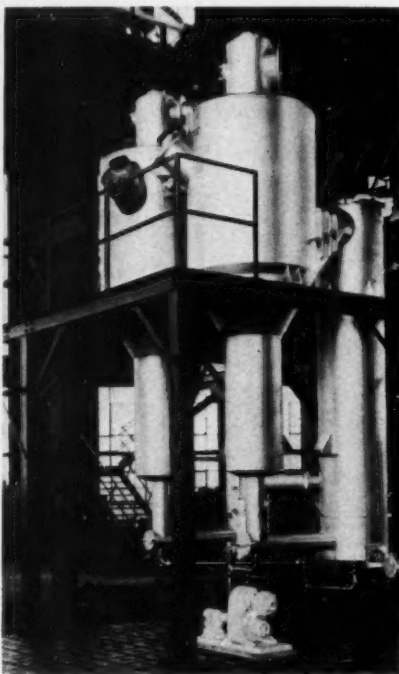
J. Schweppe and A. Foust[13] have investigated the heat transfer in a forced circulation evaporator in which boiling occurs. The tube was only 8.53 in. long and the diameters investigated were 0.622, 0.438, and 1.049 in. respectively. The effect of circulation rate on the pressure drop and heat transfer coefficient was investigated. Bulk velocities assuming liquid and vapour to be travelling at the same rate extended up to approximately 40 ft./sec. From the work it was concluded that both temperature difference, and flow rate, influence the rate of heat transfer even at the highest flow rates. It was also found that a phenomenon called sonic choking occurred at relatively low feed rates, owing to the presence of considerable quantities of vapour. Beyond this range the pressure drop decreased, this being due to suppression of boiling by the higher pressure, and then began to increase again in the way one might expect

Blaw Knox Ltd., of London, recently concluded an agreement with the Blaw-Knox Co. of U.S.A. for the manufacture in Britain of 'Buflovak' equipment for evaporating, drying, solvent recovery and other processes. The photograph shows a 'Buflovak' double-effect evaporator. The 'Buflovak' range includes long tube vertical rapid concentration, forced circulation, low temperature, downflow and thermo-recompression types, and evaporators are usually designed and supplied for a specific job. Steam chests and vapour separators are closely-coupled separate units.
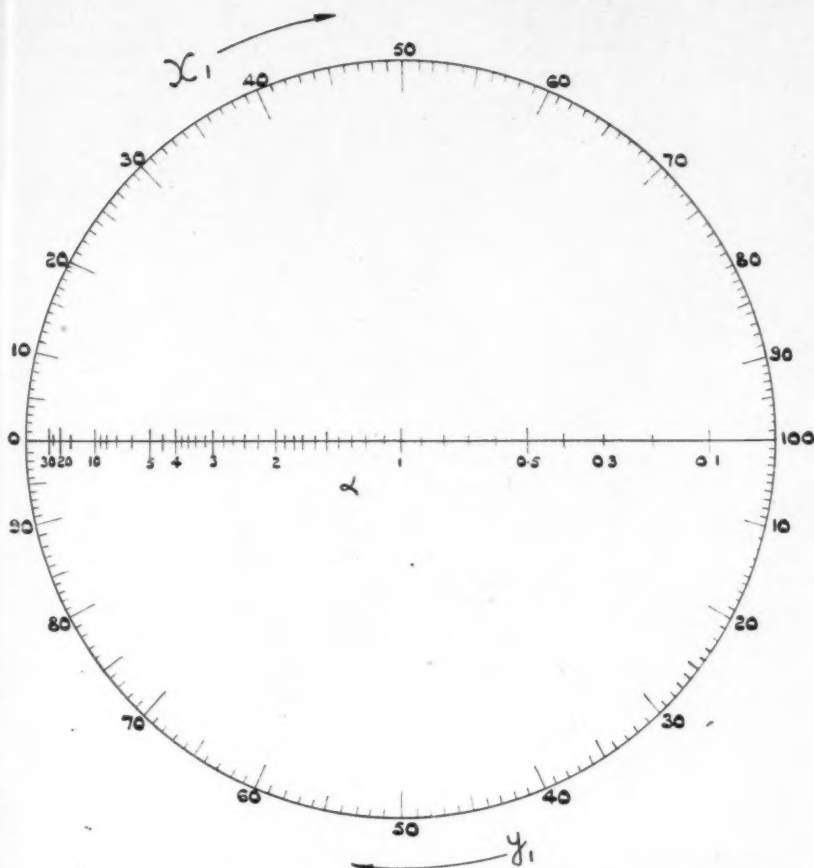
for a liquid flowing through a tube at high velocities.

Duckler and Bergalin[14] have carried out a theoretical analysis of co-current flow of a gas and a liquid film making use of the principle of a universal velocity profile. They have also carried out experimental work for a falling liquid film and stationary gas layer measuring the liquid film thickness by an electrical capacitance method. A number of photographs of the falling film are included and the transition to turbulent flow is stated to occur at 1,080.

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Cleaning in the food industry. Joseph Crosfield & Sons Ltd. have explained the application of their detergents to the food industry in a series of four pamphlets. Each pamphlet contains sections on utensils and plant, walls and floors, mechanical cleaners and vehicles and garages. These pamphlets have been produced to cover the whole of the industry, to inform manufacturers of the places where dirt is likely to accumulate, and how to use specialised equipment like the steam detergent gun. It is claimed that the most effective cleaner for the food industry is blended alkali. The effect of blending is to add properties so that problems of varying water hardness, susceptible metal surfaces and tenacious metal soiling do not affect its cleaning efficiency. The four pamphlets are: (1) Baking. (2) Canning and Deep Freeze. (3) Meat and Fish Products. (4) Sugar Confectionery and Preserves.

High-speed mills. A leaflet issued by Kek Ltd. describes the Kek high-speed mill, which is used in many industries for grinding, blending and emulsifying a large number of substances. Although it is not suitable for highly abrasive materials, most others are effectively dealt with. The design is based on the principle of horizontal high-speed centrifugal action. Apart from the driving mechanism, it consists essentially of two stout circular metal discs studded with strong metal pins set in concentric circles, openly spaced towards the centre, but gradually closing up towards the perimeter. The material is ground by being flung against these pins by centrifugal force. The suction feed is claimed to add to the efficiency of the mill.

One Nomogram Solves Several Problems

By O. P. Kharbanda, M.Sc., D.Ch.E.

(Research Department, Simon-Carves Ltd., Stockport)

LIQUID-VAPOR COMPOSITIONS	y_1 VAPOR	x_1 LIQUID	α RELATIVE VOLATILITY
WEIGHT % ⇌ MOLE %	WEIGHT %	MOLE %	M_1/M_2 (M=MOL WT)
WEIGHT % ⇌ VOLUME %	WEIGHT %	VOLUME %	d_1/d_2 (d=DENSITY)
VOLUME % ⇌ MOLE %	VOLUME %	MOLE %	$(M_1/M_2)(d_2/d_1)$

AN elliptical nomogram recently published[1] has attracted the attention of this author for two reasons: firstly, the nomogram is drawn empirically and no proof given for its construction and, secondly, this type of nomogram has not, in the past, received the attention it deserves. A circular- or elliptical-type nomogram is particularly useful when the functions of the variables concerned are such, that in a conventional-type nomogram, the graduations would lie at infinity.

Relative volatility (α) can be expressed[2] in terms of liquid and vapour compositions by means of the equation:

$$\alpha = \frac{y_1/x_1}{y_2/x_2} \quad \dots \dots \dots \dots (1)$$

where x_1 = mol. % (or mol. fraction) of the more volatile component in the liquid, y_1 = mol. % (or mol. fraction) of the more volatile component in the vapour, and subscript 2 refers to the less volatile component.

For a binary system, $x_1 + x_2 = 1$ and $y_1 + y_2 = 1$; therefore,

$$\alpha = \left(\frac{1-x_1}{x_1}\right)\left(\frac{y_1}{1-y_1}\right) \dots \dots (2)$$

solving equation (2) for x_1 and y_1 in turn:

$$x_1 = \frac{y_1}{\alpha - y_1(\alpha-1)} \quad \dots \dots (3)$$

$$y_1 = \frac{\alpha x_1}{(\alpha-1)x_1 + 1} \quad \dots \dots (4)$$

Any of the three equations (2) to (4) may be used as a basis for the nomogram. Using equation (2) and transposing α:

$$\left(\frac{1-x_1}{x_1}\right)\left(\frac{y_1}{1-y_1}\right) - \alpha = 0 \quad \dots \dots (5)$$

This is one of the standard forms, and a nomogram consisting of three intersecting straight lines[3] can easily be drawn. However, for $x = 0$ and $y = 1$, the two functions approach infinity and, for a nomogram of finite dimensions, the scales have to be considerably restricted. The same difficulty also arises if one takes logarithms of both sides of equation (2) for constructing a nomogram of three parallel straight lines. Use of a circular-type nomogram obviates such a difficulty.

The constructional determinant for equation (5) can be written by analogy:[3]

$$
\begin{vmatrix}
\dfrac{\delta_1}{1 + \left(\dfrac{1-x_1}{x_1}\right)^2} & \dfrac{\mu_1\left(\dfrac{1-x_1}{x_1}\right)}{1 + \left(\dfrac{1-x_1}{x_1}\right)^2} & 1 \\[3em]
\dfrac{\delta_1}{1 + \left(\dfrac{y_1}{1-y_1}\right)^2} & \dfrac{-\mu_1\left(\dfrac{y_1}{1-y_1}\right)}{1 + \left(\dfrac{y_1}{1-y_1}\right)^2} & 1 \\[3em]
\dfrac{\delta_1}{1 + \alpha} & 0 & 1
\end{vmatrix}
\quad (6)
$$

where δ_1 and μ_1 are the scale factors and represent, in effect, the major and minor axes of an ellipse ($\delta_1 =/= \mu_1$), or diameter of a circle ($\delta_1 = \mu_1$). The first vertical row of the determinant represents the x co-ordinate and the second row the y co-ordinate of the three scales x_1, y_1 and α. The elliptical form used[1] necessarily limits the extent of the α scale, and therefore the circular form is utilised here. The value of the scale factors is chosen as 6, to obtain a nomogram of convenient size.

To construct the nomogram, draw a straight line 6 in. long in the middle and parallel to the bottom edge of the paper and draw a circle about this line. This straight line is the x-axis and the origin is located at its extreme left-hand point. Inspection of equation (6) shows that this straight line is the locus of α (since $y = 0$), and the graduations can be marked off by

calculating $\left(\dfrac{6}{1+\alpha}\right)$ for various assumed

values of α. The arcs of the circle are the loci of x_1 and y_1, x_1 being on the portion of the circle above the straight line (y, positive) and y_1 on the lower half of the

circle (y, negative). The graduations can be marked off by calculating the respective x and y co-ordinates. The arithmetical calculations are considerably simplified by noting that only one (x or y) co-ordinate need be calculated, since the graduations must lie on the circle. Further, because of the symmetry of the co-ordinates in equation (6), the y_1 scale is easily obtained simply by joining the x_1 graduation to the centre of the circle and extending it to the far end of the circle.

The nomogram thus constructed can also be used for converting weight % to mol. %, weight % to volume % and volume % to mol. %, provided an appropriate value of α is used. Proof for this is given below for the case of volume % to mol. % conversion:

Component	Volume fraction	g./cc. density	g.	mol.
1	v_1	d_1	$v_1 d_1$	$(v_1 d_1)/M_1$
2	$v_2 = (1-v_1)$	d_2	$(1-v_1)d_2$	$(1-v_1)d_2/M_2$

Mol. fraction of component 1:

$$m_1 = \frac{v_1 \dfrac{d_1}{M_1}}{v_1 \dfrac{d_1}{M_1} + (1-v_1)\dfrac{d_2}{M_2}}$$

$$= \frac{v_1}{\left(\dfrac{M_1}{M_2}\dfrac{d_2}{d_1}\right) - v_1\left(\dfrac{M_1}{M_2}\dfrac{d_2}{d_1} - 1\right)} \quad \dots (7)$$

This equation is of the same form as equation (3) and, by comparison:

$$m_1 = x_1$$
$$v_1 = y_1$$
and $\dfrac{M_1}{M_2}\dfrac{d_2}{d_1} = \alpha$

The appropriate value of α for the various conversions is indicated on the nomogram itself. It should be obvious that any other function having variation similar to equations (2), (3) or (4) could also be evaluated by use of the nomogram.

Use of the nomogram requires simply a straight ruler. The ruler is set on the two known quantities, and the third is readily found on the third scale.

Illustrations

(a) *Calculating liquid and vapour compositions:* For the benzene-toluene system at liquid composition of 20 mol. % benzene, the relative volatility α is 2.37.[2] Set the straight ruler at x_1 equal to 20 and α equal to 2.37 and read the value of y_1 as 37.1%. By slide rule (12 in.) calculation using equation (4), y_1 is 37.2%.

(b) *Weight % to mol. %:* Consider ethyl alcohol water mixture containing 75 wt.% alcohol. Join 75 on y_1 scale with 2.55 (= M_1/M_2) on α scale and read x_1, mol.% as 53.7. The value obtained by slide rule calculation is 53.8.

(c) *Weight % to volume %:* Consider

the same mixture as in (b) above; using α of 0.795 (= d_1/d_2), 75 wt.% corresponds to 79 vol.%, which is exactly equal to the value calculated by use of a slide rule.

(d) *Volume % to mol. %:* In this case α is 3.21 (= 2.55/0.795), and 79 vol.% corresponds to 53.9 mol.%. The calculated value is 54.

Thus results obtained by use of the nomogram are of the same accuracy as those obtained by use of a 12-in.-long slide rule. The nomogram, of course, is much simpler to use and saves considerable time.

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Letters to the Editor

Changing Concepts in Chemical Engineering

To the Editor:

Dear Sir.—It is good to publish articles which discuss the 'philosophy' of chemical engineering, since there is a tendency in some quarters to be shamefaced when the subject is mentioned. Permit me to follow the publication of the article by Ross and Freshwater (C.P.E., August, pp. 231-234) with some discussion.

Analysis of the present-day activities of chemical engineers shows three principal groups, which may be termed:
(1) Experimental operation
(2) Process engineering
(3) Design engineering.

The major part of chemical engineering literature is concerned with the discussion or investigation of process engineering. This is because such matters as heat balances and the number of theoretical stages may be readily investigated by algebra and tested by relatively simple experiment. The discussion of design engineering, *i.e.* the determination of the functional shape of the reactors necessary to carry out unit steps of the production process, commenced as soon as any rational attempt was made to construct a plant, but was retarded by the difficulty of getting new information.

A pioneer in the rational analysis of chemical plant design into its functional elements was Hausbrand. More than 50 years ago he set down methods for determining the dimensions of the elements of the evaporator. I am not ashamed to have suggested a similar approach to the problems of design for fluidised process plants, which has since been followed by Sittig.

I suggest that the four groups of essential activities stated by Ross and Freshwater—handling, heat transfer, subdivision and mixing—do not express with sufficient precision the basic specification of the functional elements of any reactor or unit plant. No mention is made by them of the prime importance to design of entrainment

elimination. This functional element is frequently the limiting feature in the design of evaporators, distillation columns, leaching plant, rotary driers and fluidised reactors. I would propose to list the basic specification of a unit plant as follows:
(1) Inlets for the various phases
(2) Internal conveying of phases } These may be termed 'handling'
(3) Outlets for phases
(4) Subdivision (subsequent agglomeration may be needed)
(5) Mixing
(6) Reaction or contacting space
(7) Entrainment elimination for the various phases (*i.e.* unmixing or phase separation)
(8) Heat transfer (frequently associated with contacting space)

It may be argued that I am discussing design elements, whereas Ross and Freshwater discussed basic activities. From the side of utility I prefer the sort of specification given above for these reasons:
(1) It serves as a check for plant design.
(2) It provides a framework for the collection of existing information.
(3) It indicates the function which any test rig must perform. (The investigation of functional elements by pilot plant has recently been termed the 'unitised' approach.)

A current need is the bringing together of the principal items of information of the performance of the functional elements with the various phase systems. I hope shortly to produce a review of the field and to indicate which regions need further investigation. There is no doubt that the work to be done will provide tasks for many years for the university laboratories and, I hope, for the D.S.I.R.

S. A. Gregory, b.sc.,m.i.chem.e.
Appleby-Frodingham Steel Co.
Scunthorpe

Lessing Rings

Dear Sir.—Our good friend's (Dr. Felix Singer) review of the second edition of our textbook, 'Tower Packings and Packed Tower Design,' is so complimentary that we hesitate to complain. However, over a period of a great many years, we have yet to receive our first inquiry for ceramic Lessing rings over a 2 in. o.d. in size. You will, therefore, see that we did not make an error in stating on p. 9 that the range of usefulness of ceramic Lessing rings is limited between 1 in. o.d. and 2 in. o.d. sizes. Chemical engineers here prefer to use cross-partition and spiral rings where larger ring sizes become necessary.

Howard Farkas, *vice-president*,
The United States Stoneware Co.
New York

Fire and Explosion Hazards in Factories

PROBLEMS CREATED BY LIQUIDS, DUSTS AND PROCESS CONTROL INSTRUMENTS

IT is practically impossible nowadays to find any industry in which there is no safety problem concerning inflammable liquids and it is obvious that, as uses are found for the vast quantities of solvents and chemicals produced by the new oil refineries and petroleum chemical factories, the safety problems are going to become even more extensive and more acute.

This observation is made in the Annual Report of the Chief Inspector of Factories for 1952, which has just been published (H.M.S.O., 6s. 6d.), and in which the subject of fires and explosions due to inflammable liquids receives rather more attention than in previous years.

There has fortunately been no repetition of the type of accident that occurred towards the end of 1948 at a factory in Hull engaged in the extraction of cocoa butter from cocoa beans. (A brief reference to this accident was made in the Annual Report for 1948, where it was stated that ' Twelve persons were killed and 29 injured as a result of an escape of petrol from a process vessel. The petrol was used for the extraction of fat. A serious leak developed, petrol escaped and spread throughout the building *via* staircases and plant.')

The number of factories in which solvent extraction processes are used is, however, slowly increasing, and this increase is likely to be greater as the quantity and variety of solvents produced by the petroleum refineries and petroleum chemicals factories grows. Firms have been advised to adopt a number of basic precautions in the design and layout of these solvent extraction plants, and since the precautions apply to many other plants that contain inflammable liquids, it may be useful to state some of the principles involved.

First of all, as was illustrated all too forcibly by the Hull disaster, it is wise to keep the amount of inflammable liquid above the ground floor to an absolute minimum. Some designers of plant are apt to exaggerate the value, from the point of view of production, of gravity flow systems and to arrange plant with each successive plant unit at a lower level than the preceding one. This may mean that large quantities of inflammable liquid are in use at levels where, if there is any overfilling, failure of a plant unit or a pipeline, etc., there will be a cascade of liquid falling from a height. If the liquid is near its boiling point, or if it is volatile at room temperature, such a cascade, presenting as it will a large surface area from which volatilisation can take place, can rapidly fill the air of a workroom or of a whole building with an explosive concentration of vapour or mist. Even if the liquid is not one that will vaporise to any great extent,

the spread of the liquid, perhaps at several floor levels, introduces grave fire risks and the possibility of a ' running fire ' that is very difficult to control and extinguish. It must be remembered that in a gravity flow system the inflammable liquid has in any case to be raised initially to the highest point in the system, usually by means of a pump; the problem of transfer from one plant unit to the next when all those units containing appreciable quantities of inflammable liquid are at or near ground level is often not much greater. Sometimes an additional pump or two is required, but often one pump can be made to perform several of the transfer operations.

If it is possible to arrange the plant so that all units containing inflammable liquid are on the ground floor, it is usually a fairly simple matter to provide sills at the doorways of the rooms and to make the floor of each room a ' saucer' of sufficient volume to retain the liquid that may escape from the plant in that room. Often it is desirable to go further than this and to subdivide the floor area so that leakage from a particular plant unit or group is retained as close as is practicable to the original source of leakage. Obviously no exact rules can be laid down, but the aim should be to stop the spread of the escaping liquid before the surface area has become very large. This reduces the rate at which explosive vapours can form and, apart from risks of explosion, if a fire should occur, it will probably be confined to a limited area and consequently is very much easier to control.

When, unavoidably, plant units containing inflammable liquid are placed in elevated positions or on upper floors, means should be provided to prevent any large-scale spillage of liquid from dropping to the floors below, running down staircases, through openings in the floors, etc. The floor at each level should be made impermeable and openings in the floor should be filled in or suitably protected by low sills, etc. Subdivision of the floor area, as described above, is often desirable. In some multi-storey factories where inflammable liquids are in use the idea is prevalent that gridded or open chequer-plate floors should be used to improve ventilation and to prevent the accumulation of explosive concentrations of vapour. The ventilation of any room which permits of an atmosphere containing vapour in explosive or nearly explosive proportions is far below the standard required for healthy working conditions, and something other than the provision of gridded floors is needed. From the point of view of the prevention of fire and explosion, the best arrangement seems to be the impermeable floor system described, together with

proper ventilation arrangements at each level. Some firms suggest the drainage of floors *via* flame traps to suitable receivers in safe positions outside the factory, and this would appear to be good practice where it can be done.

Distillation of inflammable liquids

Most solvent extraction plants include one or more distillation units, and the attention of numerous firms engaged in the distillation of inflammable liquids, either in connection with solvent extraction plants or for other purposes, has been directed to the Safety Rules for the Distillation of Inflammable Liquids agreed between the Factory Department, the Association of British Chemical Manufacturers, the Association of Tar Distillers, the British Chemical Plant Manufacturers' Association and the Institute of Petroleum.

The importance of one of these rules, in particular, needs to be stressed, because neglect to observe it may result in a major fire or explosion. The rule concerns water-cooled condensers for the condensation of inflammable vapours produced during distillation processes. If there is at any time while the plant is working either no flow or an inadequate flow of cooling water, uncondensed vapours will pass the condensers and escape at the delivery end of the plant. Often it can be arranged that any such escape of vapours is *via* a vent pipe to the outside atmosphere, and where such arrangements are practicable they should always be made. Whether the escape of vapours is outside or inside a workroom, however, the resultant accumulation of a cloud of vapour which will rapidly mix with air to form an explosive mixture is obviously very dangerous. The lack of cooling water may be due to mistakes by plant operators, the cutting off of the supply at some point remote from the distillation point, blockages in the system or at the water outlet from the condenser, etc. A useful precaution is to have an audible alarm that operates, for example, either on the detection of excess temperature in the outlet from which the condensed inflammable liquid leaves the condenser or on the detection of inadequate flow of the cooling water. A better arrangement is to interlock the cooling system with the means of heating the still, so that heat cannot be applied to the still unless the condenser is working satisfactorily. Such an interlock can operate on temperature or flow in the same way as the audible alarm, and interlocking and alarm systems can readily be combined.

A simple interlock that could be more widely used is based on the ' hole in a bucket ' principle. A vessel with a hole in it is placed over an open tundish and is so

arranged that the water leaving the condenser runs into it and normally keeps it full to overflowing. The vessel is connected to a valve on the steam line to the still and the weight of the vessel full of water is sufficient to hold this valve open. If the flow of water is insufficient, the vessel empties through the hole in the bottom and is no longer heavy enough to hold the valve on the steam line open. The steam supply to the still is thus automatically cut off until the flow of cooling water has been corrected.

Fluidised flow reduces dust explosion hazard

The technical advantages of 'fluidised' systems for handling powdered materials have become increasingly recognised over the past 25 years. However, one very obvious advantage of such systems has not received as much publicity as it might; it is that the risk of dust explosions, which is so high when inflammable dusts and powders are transported by bucket conveyors and elevators, or by orthodox pneumatic processes, is almost eliminated. Cement factories use fluidised flow systems extensively, but factories in which carbonaceous and other inflammable powders are manufactured and used have been slow to take advantage of its benefits.

However, one large firm, making animal feedingstuffs, has for a number of years used this method in one part of its plant for the transport of organic meals, and has almost eliminated the danger of dust explosions in that part of the factory.

In recent years plant designers have made successful use of the system to convey catalyst, in powder form, between the reaction chamber and the regenerator. In one process in which explosive concentrations of vapour and air were difficult to avoid, a means of suppressing explosion has been made possible by the use of a 'fluidised' system. In this process the catalyst is mixed with a large quantity of powdered inert material, which produces an effect similar to that of stone-dusting in mines.

Research and development work is in progress with a view to adapting the system of 'fluidised flow' to the purification of coal gas by iron oxide. The use of such a system should substantially reduce the danger of gassing and explosion which is at present associated with the use of purifier boxes in the manufacture of coal gas.

Mechanical handling

An associated topic, also dealt with in the report, is the increased use of mechanical handling plant in industry. It is pointed out that less manual handling means fewer accidents arising from this cause, and the avoidance of heavy lifting reduces fatigue. One striking example of this is provided in the gas industry. In a reorganised works, coal and coke handling is entirely automatic by means of bucket elevators, belt conveyors and other

mechanical handling plant. The accumulations of coal and coke dust are reduced to a minimum by a vacuum ring main which encircles the plant—plug points are provided at close intervals and four men are kept continuously employed cleaning down walls and fixtures. An unusual but beneficial refinement is the provision of automatic dust extraction on the coke receiving skips at the bottom of the retorts.

Instrumentation and control in hazardous situations

The rapid increase in the use of scientific instruments in the chemical, gas and petroleum industries is introducing a new risk which has not been fully appreciated. When instruments or controllers are located in places where there may at times be a risk from inflammable gases or vapours, they should be flameproof or intrinsically safe, but a risk may also arise where instruments have been placed in an otherwise safe area, but necessarily have both gas or liquid connections and an electrical supply. The latter may be an essential part of the measuring equipment, such as the supply to a bridge network or a heater in the gas stream, or may actuate a counter or an electric clock mechanism for a recorder chart, or it may be used to relay the readings to another instrument or controller.

In such circumstances the electric circuits should be intrinsically safe or other appropriate steps should be taken to prevent an explosion inside the instrument or controller.

It is not always realised that the circuits for telephones, bells, loudspeakers and even 'deaf-aids' are not as a rule intrinsically safe and, in fact, one very serious explosion in a gas booster house was almost certainly caused by a telephone bell.

Thermit powders and dust explosions

The preparation of exothermic mixtures for the reduction of metallic oxides is a well-known process and, in general, the metallurgical firms which make use of this process are well aware of the precautions that are necessary in preparing the mixtures.

A new development in steel casting makes use of a thermit mixture to reduce the rate of cooling at the runners and risers, and considerable quantities of the mixture are being prepared for the purpose. It was found that the mixing of the thermit powders was taking place in conditions that gave rise to the possibility of a dangerous aluminium-dust explosion. The firms concerned were persuaded to adopt precautions that involved the isolation of the process from the rest of the factory and the exclusion of the workpeople from the enclosure in which the mixing was taking place. These precautions were found to be justified when, subsequently, it was learned that a very violent explosion had taken place in a German factory in which this operation was being conducted.

New Light on Hopper Design

RECENT discoveries concerning the flow of solids in bins, chutes, hoppers, conveyors, filling machines, etc., should result in better design of such equipment and speed up packaging and bagging.

It has been found that there are fewer factors governing flow than was generally thought. The key ones are:

For the hopper. Head, diameter, slope angle of sides and diameter of discharge opening.

For the material. Coefficient of friction, ratio of lateral to vertical pressure, specific weight and shear strength.

The temperature and moisture content may be indirectly accounted for or overlooked if the sample treated is representative of process material.

These facts were discovered in the materials handling laboratory of the Richardson Scale Co., U.S.A. So far three investigations have been made and completed as follows:

(1) *Measurements of pressure created by a head of material in straight- and slope-sided hoppers.* These tests were made primarily to check Jannssen's equation for pressure in circular, straight-sided hoppers and to see to what extent the equation applied to slope-sided hoppers.

(2) *Investigation of lateral and vertical pressures created by materials in different bin configurations.* Lateral and vertical pressures, under a given head of liquid, are always equal. This is not true for solids because of friction between adjacent particles and cohesion of particles.

(3) *Investigation of the mechanics of flow.* How do various materials behave when they flow from a bin? Are there any consistent patterns of flow? What effect has the geometry of the bin on discharge? What happens when material is removed from the bin while at the same time the bin is being fed?

In *Modern Packaging*, 1954, **27** (7), J. K. Rudd describes how, to study flow inside bins, a bin cross-section was built with a plate-glass front, metal back and with an adjustable discharge opening and tapering lower sides. Several studies of flow were made, using cohesive and non-cohesive materials, with various bin shapes. Results:

(1) A central column of flow exists above the discharge opening and is as wide as the opening. If material was being put into the bin at the same rate as it was being withdrawn, material outside the area of the central column consequently would remain static.

(2) Material flows into the central column area as soon as the central column is discharged.

(3) With cohesive materials, a central 'plug' moves out when the discharge gate is opened and may leave a 'rat hole.' To get further flow in this case requires rapping or hammering on the bin.

Heating Methods for Distillation Plant

By A. Pennell, B.Sc.

(Department of Chemical Engineering, University of Birmingham)

Because of its cheapness compared with other methods, steam heating is widely used in distillation plant. However, for high-temperature distillation the use of steam is impracticable because of the extremely high pressures which would have to be employed. In such cases, therefore, special heat-transfer media have to be used. Here the author discusses the various methods of heating that are available and indicates the factors to be considered in selecting a heating system for a particular distillation. The article* is reprinted by permission from ' The Birmingham University Chemical Engineer,' May 1954.

THE art of distillation has been practised for many centuries, and records show that it was even used by the ancient alchemists. Until the nineteenth century the heat was obtained from wood or charcoal fires. Early in the nineteenth century the advantages of the uses of steam were realised, and a method was introduced in which the heat was supplied by the direct injection of steam into the still.

Further developments then led to the application of heat by the use of closed steam, *i.e.* steam in jackets or coils. More recently, the need for a heat transfer medium for high temperatures has resulted in the introduction of materials such as *Dowtherm, Arochlors,* and aryl silicates. New types of electrical heating have also been developed and proposals for the use of infra-red heating have been put forward.

It is seen that the chemical engineer has a wide range of heating systems at his disposal for use in distillation plant. Since more than two million tons of coal (or its heat equivalent) are used in this country every year by distillation, it is important that the most economic method should be chosen.

Essentially distillation is a separation process and work must be supplied to a mixture in order to separate it into its components. This work is supplied in the form of heat energy, and theoretically the only energy required is that equivalent to the entropy change on mixing. In practice much more heat is required due to:
  (a) heat losses,
  (b) inefficiencies in the separating process.

In order to keep the heat requirements to a minimum these factors should be kept to a minimum. The problem of heat losses has been considered to a great extent, and Freshwater,[9] in a recent paper, has reviewed methods for the reduction of inefficiencies in the separating process.

Heat transfer to boiling liquids

Heat transfer into a distilling liquid is complicated owing to the fact that a change of phase occurs.

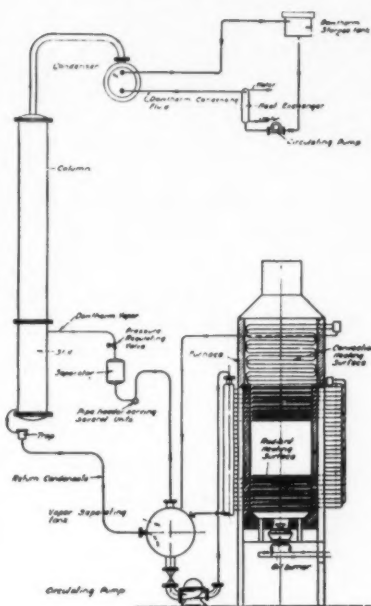The mechanism of boiling has been studied extensively and the present theory

Fig. I. Typical Dowtherm heating unit.

is that there are three distinct regions in which the boiling possesses different characteristics according to the temperature difference across the film. Two of these are defined as convective and nucleate boiling regions, while a third region is one in which meta-stable and stable film boiling occurs.

In the first two types of boiling, the heat transfer is governed by the properties of the liquid phase, but in meta-stable and stable film boiling, the heat transfer is complicated owing to the presence of the gaseous phase in considerable quantities. The presence of the gaseous phase is generally detrimental to heat transfer except in instances when it induces increased turbulence in the liquid phase.

For nucleate boiling inside a tube, Boarts[2] and his co-workers have found that the boiling coefficient varied according to the following expression:

$$h_B \propto (\mathrm{Re})^{0.8}$$

*Digest of B.Sc. thesis, June 1953.

It is probable that this relation does not hold for lower Reynolds numbers, because of the increased amount of gaseous phase.

As the heat flux (the rate of heat transfer per unit area) increases, a critical value of the temperature difference, $(\triangle t)_c$, is obtained beyond which the insulating properties of the gaseous phase hinder the heat transfer and cause the film coefficient to decrease. A gaseous blanket is formed which is unstable, collapsing and growing alternately, and is known as a meta-stable film. With an increase in the proportion of vapour, less heat is transferred until a minimum is reached, when the whole of the heating surface is covered by a gaseous film. This is the 'spheroidal state' of boiling (Leidenfrost[11] phenomenon) and any further increase of temperature will cause the heat transfer rate to increase because of increased convection across the gas film. The gaseous phase is controlling in this case, and a mathematical explanation has been put forward by Bromley.[5] Such a correlation is possible because the heat transfer occurs almost entirely through the gaseous phase, and this satisfies the usual correlation for heat transfer.

It is generally recognised that boiling inside tubes gives coefficients 25% higher than when boiling occurs outside tubes under similar conditions. Monroe, Bristow and Newell[13] postulated that for a liquid boiling outside a tube at constant temperature and pressure, the rate of heat transfer is governed by the physical properties of the fluid and the state of the surface. For boiling inside tubes, the proportion of liquid to vapour and the rate of flow of the fluid are important factors which must be taken into consideration.

The chief factors affecting the value of the coefficient are the temperature difference, the wettability of the surface, the pressure, scale and agitation.

Solid wall kettles (excluding electric heaters)

The solid wall kettle is one of the earliest types of apparatus used in distillation. The usual source of heat for such vessels is oil or gaseous fuel.

The important considerations in the

design of a kettle for transferring heat to a liquid are:

(1) the area available for heat transfer, and
(2) the rate of heat transfer.

Practical limits govern the available surface area which may be obtained. For small vessels, the use of ribs and fins has been considered, but this is impracticable in the case of larger vessels. The overall rate of heat transfer depends mainly upon the rate at which heat is transferred from the burning fuel to the vessel. The rate of conduction of heat through the wall of the vessel and the inner liquid film is usually quite high.

As in the case of pipe stills the mechanism of heat transfer is complex, but a fairly reliable indiction of the performance of a heated kettle may be obtained by use of the Wilson, Lobbo and Hottel[20] equation.

$$\mu' = \frac{1}{1 + \frac{G'\sqrt{Q/A}}{3200}}$$

where μ' is the fraction of the heat transferred.

Although the solid wall kettle is a very versatile piece of apparatus, there is a tendency for the use of more modern equipment at the present time, in order that better products may be obtained.

Electrically-heated kettles

Because of the high cost of electricity most applications of electric heating are small compared with the majority of industrial processes. Advantages of this type of heating are compactness of heating elements, the ease with which high temperatures are attained, elimination of combustion hazards and applicability for automatic control and regulation. The most common types of electric heaters are the immersion heater, the strip heater and the finned strip heater.

A more recent electric heater is the heating mantle,[17, 18] which has only been manufactured in this country since 1947, but may be used extensively in the future.

Kettles with jackets and coils

The heating of vessels by means of an intermediate heat transfer agent is often convenient for distillation at high temperatures. Certain advantages are obtained by utilising heat transfer media, such as close control of temperature and a more moderate supply of heat which eliminates the dangers of overheating. The heat transfer medium obtains its heat from an oil or gas burner and the heat is then transferred into the body of the distilling liquid. The heat transfer surface is usually in the form of a jacket or coil. An increase in the rate of heat transfer is often obtained by providing the kettles with agitators.

Heat transfer media. Steam is still the most important heat transfer medium, although many new types have recently been developed for use at high temperatures.
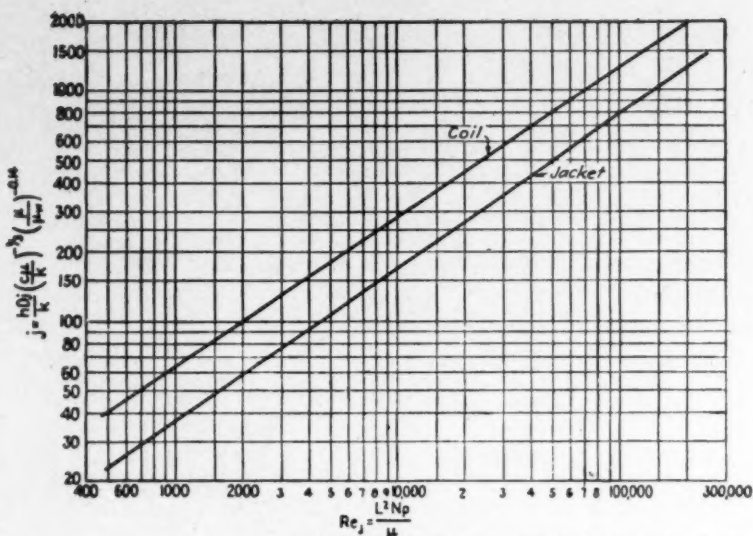
Vapours such as steam and *Dowtherm* are able to supply their heat of condensation, while liquid media such as the aryl silicates and the *Arochlors* are only able to supply sensible heat. A typical *Dowtherm* heating system is shown in Fig. 1. Liquid media, however, are extremely useful in instances where the use of steam is restricted because of the high pressures required.

Recently Brearley[3, 4] has stated that research is being carried out on the development of new heat transfer media such as *Bectherm* and *Catarex*.

Heat transfer coefficients. In 1924, Poste[15] obtained values for heat transfer coefficients in jacketed vessels by observations carried out on an actual plant. More recent work in this field has been done by Chilton, Drew and Jebens[7] who correlated data for such vessels, using the usual dimensionless groups encountered in heat transfer work, with a Reynolds number modified for mechanical agitation. Their equation is shown graphically in Fig. 2.

Cummings and West[8] extended the previous work to include film coefficients measured in larger types of equipment, which are geometrically similar to those encountered in industry. Liquids such as water, toluene, ethylene glycol and glycerol were used in the kettle and the data they obtained could be represented by an equation similar to that of Chilton et al.:

$$\left(\frac{h_j D_K}{k}\right)\left(\frac{\mu_j}{\mu}\right)^{0.14} \bigg/ \left(\frac{C\mu}{k}\right)^{\frac{1}{3}} = 0.40\left(\frac{L^2 N \rho}{\mu}\right)^{\frac{2}{3}}$$

Values for the jacket film coefficient calculated from the above equation are about 11% higher than those obtained from Chilton, Drew and Jeben's equation.

Cummings and West have also put forward a correlation for the transfer of heat

to mechanically agitated fluids by means of coils immersed in the fluid:

$$\left(\frac{h_c D_K}{k}\right)\left(\frac{\mu c}{\mu}\right)^{0.14} \bigg/ \left(\frac{C\mu}{k}\right)^{\frac{1}{3}} = 1.01\left(\frac{L^2 N \rho}{\mu}\right)^{0.62}$$

Chilton, Drew and Jebens presented a similar relation but their heat transfer coefficients were generally about 16% less than those calculated using the Cummings and West equation.

There are few data in the literature for the prediction of heat transfer coefficients inside vessels with jackets and coils, but having no mechanical agitation. For vessels of large diameter and vessels fitted with pancake coils the free convection coefficients can be calculated approximately from the equation

$$h_c = 0.38(\triangle t)^{0.25}$$

In the case of single or double helical coils, however, there is no standard method of calculating film coefficients on the outer side of the coil. However, Perry[14] gives some values of overall coefficients for coils immersed in liquids with steam as the heating medium. Overall coefficients for jacketed vessels are also given.

Individual film coefficients for heat transfer media. Barton and Williams[1] have measured the jacket heat transfer film coefficients for Merril oil, liquid *Dowtherm* and tetracresyl silicate, using forced circulation for Reynolds numbers up to 1,100. For a range of temperature difference across the fluid film from 10 to 30°C., a mean temperature of 200°C., the film coefficients ranged from 40 to 55 for *Dowtherm*, 14 to 20 for Merril oil, and 28 to 36 C.H.U./(sq.ft.) (°C.) for tetracresyl silicate.

When steam is the heating medium the film coefficient is so high that the resistance of the stream film is negligible.

Fig. 2. Heat transfer coefficients from jackets and coils.

Pipe stills

Pipe stills are used extensively in the petroleum industry, although their use is extending rapidly to other industries where crude charging stock separation is required. Gaseous fuels and oils are used almost exclusively in these furnaces, but in the future the use of by-product petroleum coke may come into prominence. Because of the scarcity and price of fuels, it has been realised that the maximum thermal economy must be obtained. The need for higher thermal efficiencies in the crude petroleum industry has led to a greater utilisation of the crude oil produced.

A pipe still consists essentially of a rectangular chamber lined with heat insulating brick, in which the tubes are located. Small heaters have burners placed in one wall of the still, but in larger types, when the heat input exceeds 20 million B.Th.U.'s per hr., heaters are usually placed in two walls. A bridge-wall made of refractory brick is erected inside the still in order to provide a heating section which is insulated from the radiation of the flame.

The part of the still bounded by the firing walls, sidewalls, roof, floor and bridge-wall, is referred to as the combustion chamber, and the heat receiving section enclosed is called the radiant section. Any heating surface behind the bridge-wall receives its heat from the hot gases of combustion before they pass to the stack. This is known as the convection section. When pipe stills were first designed they were nearly pure convection type stills, since the importance of radiant heat transfer was not appreciated. In modern stills, a large proportion of the heat transfer is in the radiant section.

Heat transfer in the radiant section. Heat transfer to cold surfaces in a combustion chamber is a very complicated process, heat being transferred in many different ways. The factors upon which the amount of radiation depends may be summarised as combustion chamber characteristics, which are not generally known in the design stage.

The heat transferred can be predicted by the Stefan-Bolzmann equation, in which the heat absorbed is related to the temperatures of the gases and tubes. However, the application of this equation would be very involved and workers such as Wilson, Lobbo, et al.,[20] have attempted to correlate data from the operation of actual pipe stills in the form of simplified empirical formulae. Scott[19] has modified the equations of these workers to some extent and has proposed the following:
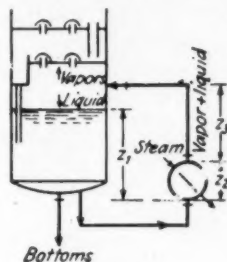
$$\mu' = \frac{1}{1 + G'\left(\frac{1.864H'}{\mu'}\right)^{0.5} \Big/ 4200}$$

μ' is the fraction of heat transferred in the radiant section.

Heat transfer in the convection section. In this section of the pipe still, heat is transferred from the hot flue gases
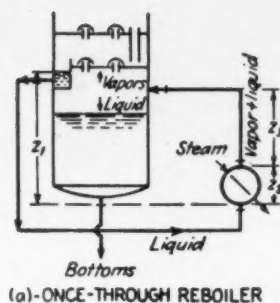
(a)-ONCE-THROUGH REBOILER

(b)-RECIRCULATING REBOILER

By permission from 'Process Heat Transfer' by D. Q. Kern, (1950). McGraw-Hill Book Co. Inc.

Fig. 3. Natural circulation reboiler arrangements.

before they pass to the stack. Heat is initially transferred by convection to the tubes, and then into the moving liquid through the metal wall. The resistance to heat flow is mainly on the flue gas side, the liquid film coefficient having little effect

NOMENCLATURE

A	Area of heat transfer surface, sq. ft.
A_C	Total outside tube area exposed to radiation, sq. ft.
C	Specific heat of hot fluid, B.Th.U./(lb.)(°F.).
D	Diameter of tube, in.
D_K	Diameter of vessel, ft.
G	Mass velocity, lb./(hr.)(sq.ft.)
G'	Air-fuel ratio
H'	Average rate of heat transfer to tube area A, B.Th.U./(sq.ft.)(hr.)
h	Heat transfer coefficient, B.Th.U./(hr.)(sq.ft.)(°F.)
h_B	Boiling coefficient, B.Th.U./(hr.)(sq.ft.)(°F.)
h_c	Free convection heat transfer coefficient, B.Th.U./ (hr.) (sq. ft.) (°F.)
K	Thermal conductivity, B.Th.U./(hr.)(sq.ft.)(°F./ft.)
L	Length of paddle, ft.
N	Number of revolutions per hour
Q	Rate of heat transfer, B.Th.U./hr.
R	Dirt factor, (hr.) (sq. ft.) (°F.)/B.Th.U.
T	Absolute temperature, °R.
$\triangle t$	Temperature difference, °F.
U_C, U_D	Overall coefficient of heat transfer, clean coefficient and design coefficient respectively, B.Th.U./(hr.)(sq.ft.)(°F.)
Z	Static height, ft.
μ	Viscosity, lb./(ft.)(hr.)
μ'	Fraction of heat transferred.
ρ	Density, lb./cu.ft.

Subscripts:

c	Coil	K	Kettle
j	Jacket	W	Wall

upon the overall heat transfer coefficient. Overall coefficients generally range from 2 to 10 B.Th.U./(sq.ft.)(hr.)(°F.).

Several equations are available for calculating the heat transfer coefficient on the flue gas side. Reiher[16] put forward an equation based upon dimensional analysis and Chappell and McAdams[6] presented a well-known equation which has since been found to be dimensionally unsound. Monrad[12] proposed an equation based upon available data:

$$h_c = \frac{1.75 \ G^{\frac{2}{3}}T^{0.3}}{D^{\frac{1}{4}}}$$

Reboilers

In certain types of distillation plant, heat is supplied in an external heat exchanger, usually by the use of steam. Reboilers fall into two main classes, those employing forced circulation and those using natural circulation.

In a forced circulation reboiler arrangement the pump circulates the liquid through the reboiler as many times as it is economically permissible, in order that the percentage vaporised each cycle is kept to a minimum. Generally, forced circulation reboilers are only used for small installations, or when the bottoms liquid is so viscous and the pressure drop through the reboiler and fittings is so high that natural circulation is very difficult. Natural circulation reboilers are most commonly used. The liquid is caused to circulate by hydrostatic pressure. Such reboilers may be either once-through reboilers, or recirculating reboilers, as shown in Fig. 3. In a once-through reboiler the liquid is only circulated once, but in recirculating reboilers the liquid is free to circulate as many times ans the hydrostatic pressure difference $(Z_1 - Z_3)$ will allow. The usual minimum recirculation ratio is 4 : 1.

A comparison of the relative merits of forced circulation and natural circulation reboilers is concerned partly with the economic aspects and partly with the space available. Forced circulation reboilers require use of a pump with its continuous operating charges and fixed cost. However, more space is required for a natural circulation arrangement and the circulation rate cannot be controlled as readily as for forced circulation.

It is obvious that the pressure drop through a natural recirculating reboiler must be kept as low as possible, in order that the hydrostatic head will be sufficient to overcome the pressure drop and also provide a circulation through the reboiler. If the pressure drop is high, the column and its auxiliaries must be lifted above the ground, in order to obtain sufficient hydrostatic head to overcome the pressure drop. In some instances the reboiler has been set in a well but this is not a common practice in modern plants.

Maximum heat flux and critical temperature difference. The phenomenon of vapour blanketing which has been pre-

viously described, constitutes a serious problem in the design of reboilers. Care must be taken that the critical temperature difference is not exceeded while the reboiler is in operation. The maximum flux is obtained at the critical temperature difference, but beyond this value the flux decreases.

The flux is defined by Q/A or $U_D \triangle t$ where U_D is the design coefficient of heat transfer. Since the flux is based upon the acutal surface area A, in which the dirt factor has been taken into account, the value of $h_v (\triangle t)_w$ does not represent the flux because this flux is based upon the clean area A_c. Usually both the flux Q/A and the vaporisation coefficient h_v are restricted, in order that the temperature difference does not exceed its critical value.

Kern[10] gives the following restrictions for the values of the flux and film coefficient:

(1) The maximum allowable flux for forced circulation reboilers using organic liquids is 20,000 B.Th.U./ (sq.ft.)(hr.), and for natural circulation reboilers it is 12,000 B.Th.U./(sq.ft.)(hr.).

(2) The maximum allowable vaporising film coefficient for forced or natural circulation using organic liquids is 300 B.Th.U./(sq.ft.)(hr.)(°F.).

The use of very high temperature differences is not advantageous however, since at the maximum allowable flux any increase in $\triangle t$ must be offset by a decrease in the value of U_D. High temperature differences are only justified when U_D is naturally small. The maximum value of U_D which is obtainable is given by $(Q/A)(1/\triangle t)$. When fixing the temperature of the heating medium it is noted that the use of a large temperature difference decreases U_D, which in turn gives a large dirt factor $R = 1/U_D - 1/U_C$.

If the temperature of the heating medium can be selected independently as in the case of steam (by the use of pressure) it is not necessary to have a higher value of $\triangle t$ than that which gives a design coefficient U_D corresponding to the desired dirt factor.

Conclusions

Steam heating is widely used in distillation plant, chiefly because of its cheapness compared with other methods. However, for high temperature distillation, the use of steam is impracticable because of the excessively high pressures which would have to be employed. At such high temperatures heat transfer media such as *Dowtherm* vapour, *Arochlors* and aryl silicates are used.

The selection of a heating system for a particular distillation is governed mainly by the following factors:

(1) **Capacity of the distillation unit.** The throughput of a distillation unit is often the limiting factor in deciding whether or not the use of a reboiler is possible. Electric heating is restricted to small units.

(2) **Temperature of the distillation.** For temperatures up to 400°F., steam is

widely used. Mineral oils and liquid *Dowtherm* are also used in this range. As previously pointed out, *Arochlors*, *Dowtherm* vapour and aryl silicates are utilised for higher temperatures.

(3) **Nature of the distilling liquid.** If the fluid being distilled is classified as a dirty fluid, it is advisable to use jackets in preference to coils, since deposits on coils are much more difficult to remove. Also if the liquid is inflammable, it is necessary to use a heating system which minimises the risks due to fire.

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Chemical Research in Russia

THE number of chemists and chemical technologists in the Soviet Union has greatly increased in recent years following the separation of the chemistry faculties of universities from those concerned with mathematics and general science. According to an article in the East German journal, *Chemische Industrie*, of June 1954, there are now tens of thousands of qualified chemists working in industry and research. There are more than 80 chemistry faculties and institutes.

The chemistry section of the Soviet Academy of Science comprises 70 members and several thousand scientists and runs 11 research institutes. In addition there are many specialised research institutes and laboratories in the chemical industry itself.

Thousands of chemists are also working in other branches of industry, *e.g.* in the metallurgical and machine construction industries, power production, etc.

Major tasks

Among the major fundamental tasks confronting Soviet chemists are the further development of Butlerow's theory of structure; study of the laws governing chemical reactions; research on the theory of catalysis; the discovery of more efficient catalysts; research on polymerisation, etc.

In many cases, Soviet scientists have been able to improve industrial processes. It is claimed, for instance, that the consumption of coke per ton of cast iron has been reduced from about 1.1 to 0.8 tons. Production of sulphuric acid per chamber has been increased more than 10 times since 1935 and superphosphate production per unit has been increased eight times.

An objective collectively pursued in

recent years has been the elimination of waste, *i.e.* the reduction in the quantity of unwanted by-products and the greatest possible utilisation of desired or unavoidable by-products.

Among the new processes developed by Soviet chemists in connection with the five-year plans are the butadiene synthesis of Lebedew; the petroleum cracking process of Shuchow and Kapeljuschnikow; the aromatisation of hydrocarbons by the process of Selinsky, Moldawsky and Kasansky; and the synthesis of camphor by the Tischtschenko process.

In the field of radiation research, the Radium Institute, founded by Chlopin in 1922, has developed radium production from raw materials available in the country and has carried out intensive research on cosmic rays.

Soviet chemists are being called on to help increase food production by developing fertilisers and crop protection chemicals. Fertiliser output by 1964 is planned to be six times that of the 1950 rate. Weedkiller output is to be doubled and the quality of the products improved. A good deal of research is to be done on insecticides, fungicides, growth-promoting chemicals, etc.

Another topical task is the development of synthetic rubber production, based on petroleum gases, synthetic alcohols and other organic substances.

A great expansion is predicted in the fields of plastics, synthetic fibres, dyes, paper production and petroleum technology. Much attention is being paid to the extraction of liquid fuels from shale, to the development of the medico-pharmaceutical industry, etc.　　　　E.R.

Comparative Efficiencies of American

BEET SUGAR REFINERIES—2

In the first part of this study of beet sugar refining in the U.S., which appeared in our August issue, the operations in a typical plant were described. In this second and final part, plant characteristics for six representative beet sugar refineries are given for comparison. Also described are the ion exchange process of juice purification, the Steffen sugar recovery process, the recovery of monosodium glutamate, and finally there is a note on production control.

Details of various plants

PLANT A is the smallest refinery of the examples under review, having a slicing capacity under 2,000 tons per 24 hr. and employing 173 production workers. Statistics on production, quality of beets and length of campaign may be found in Table 5 (see August issue, p. 249).

The plant is one of many under the control of a parent company, as are all other plants in the survey. The parent company performs such functions as administration, sales and purchasing, in addition to research, standardisation of chemical analysis and production control.

Research at the plant is directed towards reduction of lime usage, better waste disposal, less scaling, and by-product development. Improvements have been made in methods of trash, mud and rock handling to overcome problems created by mechanical harvesting. Limitations of the process equipment in handling mud or adobe, however, is one factor controlling the slicing capacity of the refinery.

Higher man-hours per ton than other plants are also shown for the diffusion operation as a result of the older batch-type process. The weight of the cossettes entering diffusion is calculated from the weight of juice and per cent. of sugar in juice, pulp and cossettes, using a raw juice scale developed by the company.

Pulp press water is not used in diffusion, nor is an antifoaming agent. Both activated carbon and filter aid are added before thick-juice filtration. The plant has 11 centrifugals which are loaded and discharged manually by four men, while all parts of the cycle are automatically controlled.

All sugar is packed, the packing department operating continuously during the campaign. Packaged sugar is conveyed by inclined belt elevators to the stacking level in the warehouse, which is air-conditioned to maintain 60% maximum humidity.

Plant B is of medium size with a slicing capacity of about 3,000 tons per 24 hr. It is the newest plant in the list, and as such has more equipment of later design than the other refineries.

Equipment features in this plant include Silver chain-type continuous diffuser, automatic pH and evaporator controls, automatic carbonation end-point control, and
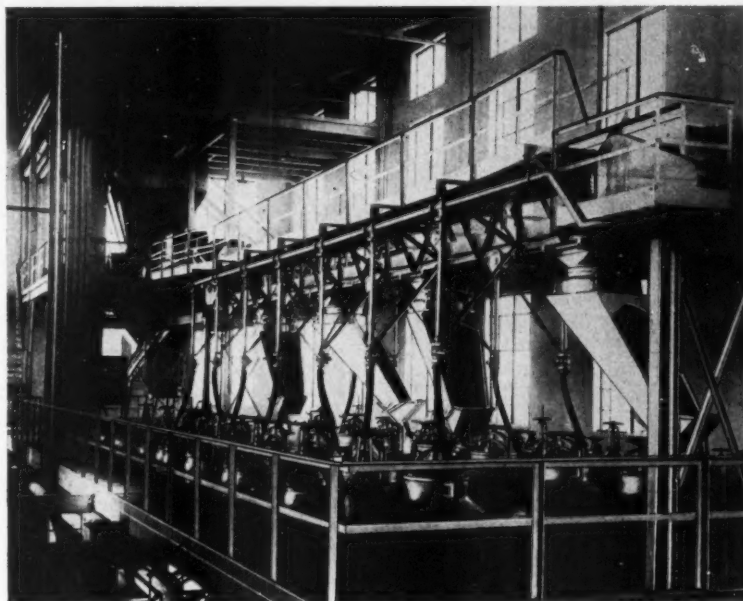
Conventional diffusion battery station with cossette conveyor and distributors.

automatic (except discharge) centrifugals.

Research at the plant includes methods of disposal of pulp press water, which is being used in diffusion, and laboratory-scale study of the ion-exchange process. Beets are stored at this plant, as at most of the other plants, in piles over metal ducts with openings through which blowers force air for ventilation.

No antifoaming agent is necessary because of high-purity beets. First, carbonation juice is filtered on rotary vacuum filters, which require about half an hour for cleaning every 8 hr. Only three of the four filters are used at one time. The cloths of the plate and frame presses at second carbonation require about 2 hr. for cleaning every 24 hr.

As in most plants, waste water goes to a pond and is later discharged into rivers. Waste lime cake is also pumped to a small pond.

Plant C also has a daily slicing capacity of about 3,000 tons per 24 hr. Beets are generally received by truck, stored in unventilated sheds, and carried to the factory by open flumes. Eighty-five per cent. of

the flume water is recirculated, whereas between 50 to 60% is recirculated at plants A, E and F. Plants B and D reported none. Handling man-hours were high in 1951 because many beets were received by rail and required piling.

Seventy per cent. of the sugar content of the molasses from regular refinery operations is recovered at this plant by the Steffens process. The waste from this process is concentrated and sold for the recovery of monosodium glutamate.

Almost half the sugar produced during the campaign is stored in bulk for packaging between campaigns. This enables the packing, warehousing and shipping operations to be performed on only one shift, and eliminates the need for precise estimates of amounts and types of packages to fulfil consumer requirements.

The company indicates that for this plant the labour cost (wages paid to production and related workers, excluding administrative, sales and office personnel) comprises 25% of total manufacturing cost, and materials 24%.

Plant D was erected in the middle

1920s, has a slicing capacity of more than 2,000 tons per 24 hr. and has adopted many of the automatic controls more recently developed. However, it is concluded that the cost of major equipment replacements has prevented this refinery (and others erected before 1940) from installing certain improved methods such as continuous diffusion. As at most of the other refineries, however, the beet piles are ventilated and the warehouse is air conditioned. No utility men were reported for this plant. At other plants such employees work at different operations to relieve regular operators for short rest periods.

Plant E has a slicing capacity of more than 3,000 tons per 24 hr. It has continuous diffusion, many automatic controls, and facilities for mechanised handling and shipping of bulk dry pulp and bulk white sugar. A detailed description of the operations of this plant, whose operations required the least number of man-hours per ton of the surveyed plants, is given earlier in this report.

Plant F is the largest refinery of the series. Although the plant has up-to-date continuous diffusion system and also continuous carbonation, much of its equipment is more than 40 years old.

This older equipment, particularly filters and presses, accounts for much of the higher man-hour requirements of this plant. The man-hours devoted to cleaning of filter cloths per 24 hours have been enumerated as follows: six man-hours for four vacuum filters, 10.5 man-hours for one large Kelly press, 18 man-hours for two Sweetland presses, 78 man-hours for six plate and frame presses at first carbonation, 81 man-hours for six plate and frame presses at second carbonation, and 81 man-hours for six plate and frame presses for thin and thick juice carbonation.

Lime cake, which is a waste product at other plants, is processed at this plant for the recovery of lime. During the 1951 campaign, almost 5,000 tons of lime were recovered, reducing requirements of coke by almost 600 tons. The pulp from diffusion is pressed and dried at this plant.

The water from pressing, which is also a waste product at all other plants except plant B, is clarified and used in the Steffens operation to dilute molasses. Approximately 2.1 additional tons of sugar is recovered from every 1,000 tons of pressed pulp water sent to the Steffens process.

Machinery and equipment

Complete information on machinery and equipment was not available from all plants in the sample. However, similar equipment was employed by all plants with few exceptions such as diffusion and filtering.

Information concerning the installation of equipment in more recent years was not available. Such installations, however, have emphasised more automatic operations. This is evidenced by continuous diffusion systems with automatic cossette scales
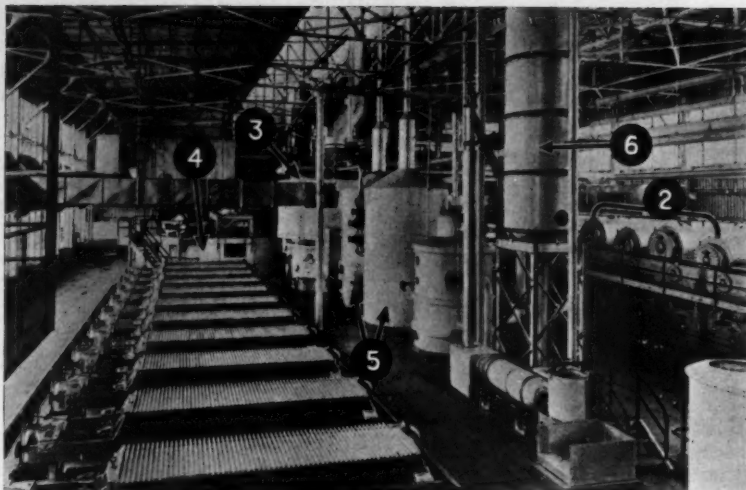
Processing units at Plant B, including silver continuous diffuser, Bennin carbonator (2), Dorr thickener (3), Shriver plant and frame presses (4), second carbonation tanks (5) and sulphur tower (6).
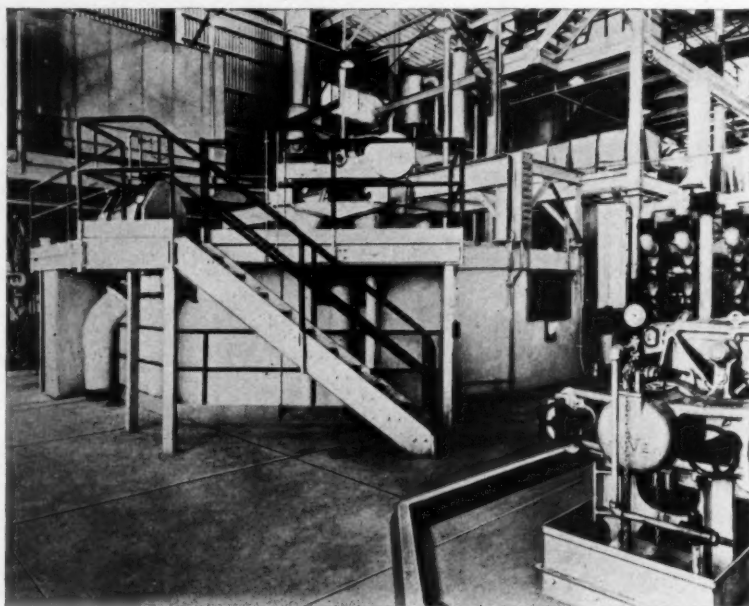
Dorr multifeed carbonation thickener.

and controls; numerous automatic level, temperature, and pH controls; and almost completely automatic centrifugals. Batteries of six centrifugals can now be operated by only two men.

Other additions have provided facilities for handling bulk dry pulp and for the handling and distribution of bulk granulated and liquid sugar. Better packing methods feature the valve-type bag which requires no gluing or sewing.

Research efforts are producing results in reducing the use of lime and filter aids, reducing foaming and scaling, and improving waste disposal and by-product recovery. Increased sugar extraction has been obtained by the return to process of pulp press water and molasses, and from the ion-exchange process.

Ion exchange process

The ion-exchange process of juice purification has been tried or proposed for treating diffusion juice, second carbonation juice, green syrups, molasses, and Steffens filtrate. At present, three beet-sugar refineries are equipped to use this process on second carbonation juice.

In the process, the juice flows through beds of resins of exchangers which remove practically all the salts and many of the organic impurities from the juice before it is boiled. The advantages claimed for this process over conventional methods

Factor	Type of operation	
	Conventional	Ion exchange
Daily slice		
Cossette sugar, tons	1,600	1,600
Sugar entering battery, tons	16	16
Beet end losses, tons	256	256
Beets: %		
Pulp 0.13		
Water 0.10		
Lime flume 0.02		
Unknown 0.15		
Total 0.40	6.4	6.4
Net sugar in thin juice, tons	249.6	249.6
Ion exchange loss including sweetening off and inversion, tons	..	6.0
Sugar to sugar end, tons	249.6	243.6
Purity, thick juice	90	96.5
Purity of molasses	60	60
Sugar-end extraction, %	83.3	94.5
Sugar bagged, tons	207.9	230.2
Sugar in molasses, tons	41.7	13.4
Molasses at 50 polarisation, tons	83.4	26.8
Extraction, % on sugar in beets	81.2	89.9
Costs	$	$
Labour, two men per shift	..	60
Regenerants per ton beets:		
Acid, 20 lb. at 0.015 lb.	..	480
Ammonia, 6 lb. at 0.05 lb.	..	480
Fuel, 1% additional	..	10
Sugar bags, 446 bags at 0.16	..	71
Total costs	..	$1,101
Credits:		
Extra sugar, 446 bags at $6.50	..	2,900
Decreased molasses, 56.6 tons at $20 ton ..	1,132	
Credits minus costs		667
Return per ton beets		0.41

Quintuple-effect, calandria-type evaporator.

include increased yields of granulated sugar, with reported extractions up to 93%; higher purity; better colour removal; lower ash content; crystallisation of two or three ' strikes ' of white sugar instead of one; elimination of evaporator and pan boil-outs; fewer processing problems; production of edible molasses; and greater potentiality for by-products.

Disadvantages which have limited the wider adoption of the process are the high initial cost of new equipment, the high costs of the resins and regenerating chemicals, and costs of transportation for the materials.

The equipment of the surveyed plant includes four pairs of tanks with heads designed for a working pressure of 60 p.s.i. The tanks, pipes and valves are rubber-lined for protection against acid. The process is controlled by the push-button operation of switches which open or close the valves. Table 6 indicates the increased extraction of sugar using the ion-exchange method and the dollar return from the extra sugar. Costs which are the same for either method used were excluded from the cost balance.

Steffens process

Although European beet-sugar refineries do not try to recover additional sugar from beet molasses because of its value as a source of alcohol, yeast and stock feed, this practice, called the Steffens process,* is fairly common in the United States. It consists of adding powdered dry lime (CaO) to the cold (about 12°C.) and diluted (about 6% sucrose) molasses syrup under agitation.

The lime combines with the sugar to form tricalcium saccharate which precipitates and is filtered. The filtrate is heated and the precipitate removed by settling and filtration. The final filtrate serves as a raw material for the recovery of by-products.

The two filter cakes are dispersed in about 10% refinery sweetwater, heated and introduced into the factory carbonation with the diffusion juice, replacing the milk of lime used in conventional refineries. The heated saccharate then decomposes to sugar and calcium hydroxide, which serves as the purifying agent for the diffusion juice. In plants where the Steffens process is used the total recovery of sugar from beets sliced is almost 90%.

In one refinery in the United States, the Steffens discard-molasses is subjected to the barium saccharate process, and rotary kilns are used for barium regeneration. The world's principal source of raffinose is crystallised directly from the final molasses in this plant.

The Steffens filtrate, although comprising only about 4% of total refinery waste

*Information on the Steffens process and monosodium glutamate, as well as much valuable background and processing data, was obtained from R. A. McGinnis (*Beet Sugar Technology*, Reinhold Publishing Corporation, New York, 1951, pp. 135, 477, 481).
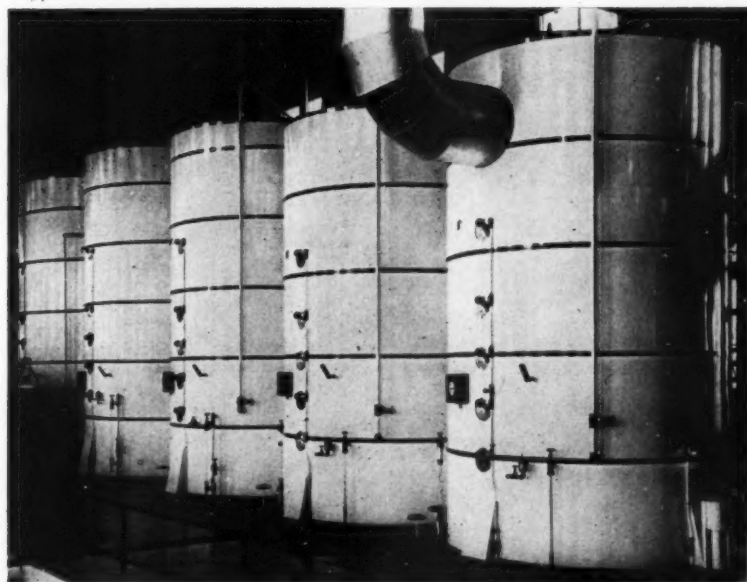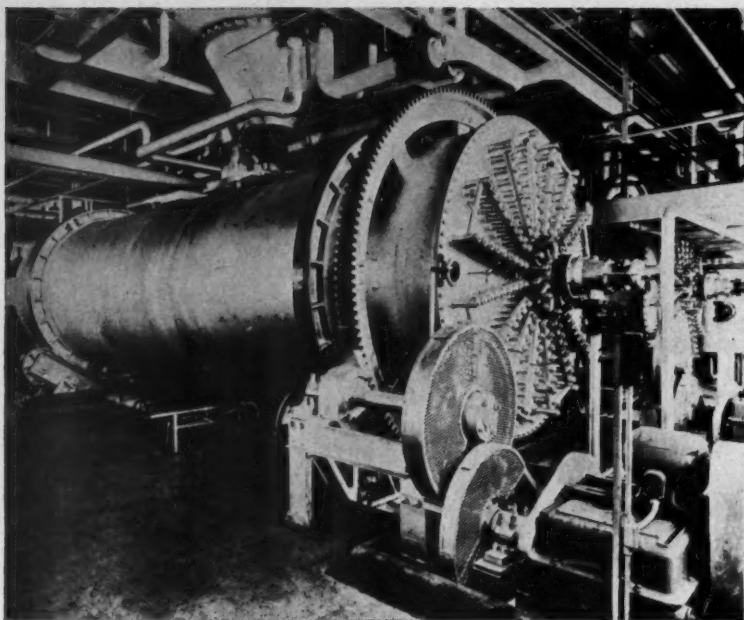
La Feuille rotary crystalliser.

volume, is responsible for almost half the biochemical oxygen demand, and as such creates the serious problem of disposal. Some refineries have installed equipment which carbonates and concentrates the filtrate, which is then sold to manufacturers of amino acid products.

Monosodium glutamate

Several plants recover monosodium glutamate,* a flavour-accentuating agent, by alkaline hydrolysis of the concentrated Steffens filtrate. After hydrolysis of the filtrate with a 50% solution of caustic soda, the liquid is partially acidified with hydrochloric acid and again concentrated.

The mixture of hydrochloric and glutamic acids creates a difficult problem of metal corrosion which requires the use of stainless-steel or rubber-lined equipment. The concentration results in the crystallisation of inorganic salts which are removed by centrifugal action.

The filtrate is further acidified and the solution crystallised, the latter requiring from five to eight days; the crystals are separated by high-speed centrifugals. The filtrate may be processed to obtain betaine. The crude glutamic acid is then purified, concentrated, crystallised and centrifuged, producing pure (almost 100%) mono-sodium glutamate.

Production control

The control of production of granulated and liquid sugar, pulp, molasses, process materials and by-products is of particular importance in the industry, not only to supply a cumulative accounting record of output but also to furnish comparative data for analysis of costs and efficiency. The examination of such data from all

*R. A. McGinnis, loc. cit., pp. 135, 477, 481.

plants is one means of effective control by the parent company over the operations of its many plants.

The continuous operations of the refinery, with three 8-hr. shifts daily, throughout the short campaign period, require numerous hourly checks on all phases of operations. It is essential that the process continue without interruption and with as high an extraction of sugar as possible, because beets are received continuously from the fields and prolonged storage reduces their sugar content.

The smallest number of man-hours expended per ton of beets is always associated with the fastest slicing rate, because the workers, in most cases, perform the same functions for any slicing rate. The data which indicate the performance of the various operations are recorded by the foremen, chemists' samplers or operators on forms ad hoc.

Such records show both operating characteristics of the equipment and chemi-

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Contributions and Correspondence

THE EDITOR welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to THE EDITOR, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.

Letters for publication on any of the subjects covered by this Journal are also invited.

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cal or analytical results of tests on the material in process. In addition, a major control item is the listing of delays and their causes.

New British Standards

Testing chemical stoneware (B.S. 784: 1953, 3s. net). The revision of this standard comes 15 years after its first publication. Since then certain of the tests specified have been found to be of academic interest only and others have been superseded by newer tests. The new document gives test methods, with full details of apparatus, test piece and procedure for determining: resistance to compression; transverse strength; resistance to abrasion; true specific gravity; apparent specific gravity, apparent porosity and water absorption; thermal expansion; resistance to thermal disruption; acid soluble iron; and resistance to acid.

Conductive and anti-static rubbers (B.S. 2044: 1953, 2s. 6d. net). This covers laboratory tests for resistivity of conductive and anti-static rubbers. Rubber is normally regarded as a material of high electrical resistivity; consequently it is widely used as an insulator. However, the incorporation of various materials, in particular certain forms of carbon black, reduces the electrical resistance greatly so that the volume resistivities between 10^{15} ohm-cm. and 1 ohm-cm. are obtainable. There are various technical and industrial purposes for which rubber with a reduced resistivity is a useful material, the most frequent application being for the dissipation of static charges.

Products which, while conducting away static charges, are sufficiently insulating to fulfil the safety requirements above are termed 'anti-static' rubbers. Products which do not fulfil the safety requirements are termed 'conductive' rubbers.

Certain types of electrode, when applied to these rubbers, have a contact resistance which may be many thousand times greater than the intrinsic resistance of the test piece. Dry contacts under light pressure or point contacts are particularly poor. The definition of a suitable electrode system is therefore an important part of this specification and, in order to satisfy the various practical requirements for tests on laboratory-prepared test pieces, a variety of suitable electrode systems is given.

Selection of fits (B.S. 1916, Part 2: 1953, 7s. 6d. net). This guide provides recommended tolerances which can be used for a wide variety of standard applications. The guide is fully illustrated and includes much useful information on the various grades of fit, the accuracy which can reasonably be expected from various manufacturing processes, etc. It also contains tables of preferred fractional and decimal inch sizes, the use of which is indispensable to true standardisation.

Viscometry of Gases at
# High Temperatures and Pressures

Accurate data on the viscosity of gases are required in designing transmission and measurement systems for fuel gases and in constructing gas-flow systems in chemical plants. A new device which makes it possible to obtain hitherto unavailable data on the viscosity of pure gases and their mixtures has been designed by the U.S. National Bureau of Standards and is described in the following article.

TO provide data for the design of gas turbines and jet engines, the U.S. National Bureau of Standards is conducting a broad programme of research on the viscosity of gases at elevated temperatures and pressures. Under the sponsorship of the U.S. National Advisory Committee for Aeronautics, hydrocarbon mixtures and combustion products composed of various proportions of nitrogen, carbon dioxide, argon, oxygen, carbon monoxide and water vapour are being studied. In the course of this work, J. C. Westmoreland, of the Bureau's staff, has developed a unique type of flowmeter which accurately controls and measures the flow of very small volumes of gas, such as those required in a small capillary tube viscosimeter. As a result of this development, it has been possible to obtain data on the viscosity of pure gases and their mixtures hitherto unavailable.

Accurate data on the viscosity of gases are required in designing transmission and measurement systems for fuel gases and in constructing gas-flow systems in chemical plants. Because the viscosity of a gas is an important factor in the rate of heat transmission between gases and metals, gas-viscosity data are also needed in the design of gas turbines and jet engines. With the continued development of new engines of this kind, the need for information on the viscosity of the exhaust gases at high temperatures is becoming increasingly important.

At present, available data on the viscosity of gases are very meagre, particularly at temperatures above 1,000°F. To meet this need, the Bureau has undertaken to determine the viscosity of pure gases and gas mixtures at pressures from 5 to 10,000 p.s.i. and over the temperature range from 60 to 2,500°F.

Viscosities are measured by timing the flow of the gas under study through a very fine capillary tube. If the dimensions of the tube are known, the viscosity can be computed by means of Poiseuille's law from the volume rate of flow through the tube and the pressure drop from inlet to outlet of the tube. The capillary tubes are of Inconel, with average internal bores of 0.038 cm. and lengths up to 434 cm. For high-pressure operation the tubes are enclosed so that they have only to withstand the small pressure differential caused by the flow of gas. The capillary enclosure is heated electrically, and temperatures are
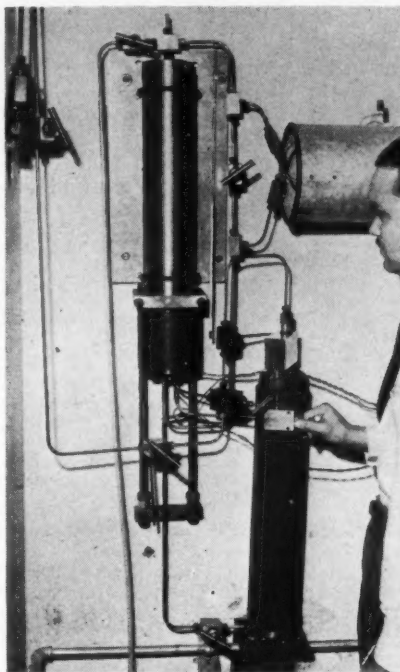
Apparatus recently developed by the U.S. National Bureau of Standards for study of the viscosity of gases at high pressures. A pressurised model of the new flowmeter (within the two vertical cylinders) is used to measure the rate of flow of the gas under study through a very fine calibrated capillary tube enclosed in the insulated and heated enclosure at upper right. The pressurised container at upper left contains all of the flowmeter except the gas-mercury reservoir, which is in the pressurised container below and to the right of the other.

measured with chromel-alumel thermocouples. At high temperatures a coiled capillary is used.

To obtain as accurate viscosity values as possible, it was decided to measure the rate of flow of the gas through the capillaries by means of some metering device employing a volumetric displacement. This method would have the advantage that the volume could be evaluated directly by conventional methods and would not involve the measurement of an auxiliary quantity. As no flowmeter capable of measuring the extremely small flow rates through the tubes was available, it was necessary to design such an instrument. The apparatus that was developed not

only determined these flow rates accurately at pressures up to 500 atm., but also includes a means of regulating the rate of flow and of measuring or pre-establishing the pressure drop over the capillary.

In previous investigations determination of the viscosity of a gas at high pressures has required a knowledge of the compressibility effects of the particular gas being tested. However, in the present experiments the new flowmeter is used to measure the volume rate of gas flow at the static pressure of the test, and an absolute knowledge of the compressibility of the gas may not be required. Under the conditions of operation of this device, the Poiseuille expression is completely defined at the test pressure and temperature, and the relationship is essentially independent of the thermodynamic deviations from the ideal gas.

The new flowmeter

The flowmeter developed at the Bureau consists essentially of a calibrated volume chamber, a float-valve chamber and a gas-mercury reservoir. These are connected in with the calibrated capillary tube to form a closed system in which the gas under study flows out of the reservoir and through the capillary tube to displace mercury in the volume chamber. The float valve regulates the flow of mercury out of the volume chamber as it is displaced by the incoming gas, and at the same time maintains constant the height of a mercury column which provides a hydrostatic pressure causing the gas to flow through the capillary tube. As the mercury passes out of the volume chamber, it breaks electrical contact with successive platinum electrodes, thus activating relays which start and stop a clock. In this way the time required for the measured volume of gas to flow through the capillary tube is obtained. No energy from the gas stream is employed in replacing the mercury, which is made to flow by gravity out of the calibrated container through the float valve chamber at a rate equal to the rate of gas inflow. The replaced mercury is then added to the top of the mercury in the hydrostatic column, replacing mercury which passes out of the small-diameter tube forming the power part of the column and into the gas-mercury reservoir. The mercury which thus enters the reservoir in turn replaces the gas which is con-

tinually leaving the reservoir to pass through the calibrated tube. A pressure-equalisation tube which joins the calibrated gas collection chamber to the float valve chamber ensures that the pressure differential across the capillary tube shall be only that due to the column of mercury above the gas-mercury reservoir.

The volume chamber is made of glass and is divided into two equal parts, one above the other, with a necked section in between the two and at the base of the lower chamber. The principal purpose of the upper volume is to provide a starting period during which flow conditions may become stabilised. The necked sections make the cross-sectional area small in the plane of the contact break.

The float chamber, float and valve are all made of methyl methacrylate. It was found that a valve and valve seat of this plastic are not eroded by the flowing mercury as a steel valve would be. Moreover, the mercury remains clean in contact with the methyl methacrylate.

In the operation of the flowmeter, the entire apparatus, with all of the mercury in the reservoir, is first connected to a high-vacuum pump and purged of all gas as completely as possible. A valve in the column between the float-valve chamber and the gas-mercury reservoir is then closed, and a gas-sample inlet valve is opened, admitting gas from an external container to the reservoir. This causes the mercury in the reservoir to rise through an auxiliary connecting tube into the passage above the float valve. As the tube below the float valve is now blocked off, the mercury will first fill the float chamber enough to close the float valve and will then fill the calibrated volume chamber. A cock in the auxiliary connecting tube is then closed. When the inflow of the sample gas has brought the pressure within the reservoir to the desired value, the gas-sample inlet valve is also closed.

Before measurements are taken, sufficient time must elapse for pressure conditions to equalise throughout the system. Then the meter is placed in operation by opening the valve in the column connecting the float-valve chamber with the gas-mercury reservoir. The outflow of mercury from the float-valve chamber lowers the float, opening the float valve so as to admit mercury from the calibrated volume container into the float-valve chamber. Whenever this inflow from the volume chamber exceeds the outflow into the reservoir, the float valve automatically closes. Thus the pressure which the mercury column exerts on the gas entering the capillary tube is kept constant, and the rate at which mercury flows out of the volume chamber is controlled by the rate of flow of gas through the capillary.

In making the determinations, the apparatus is first pressurised at a specific temperature. Flow measurements are then carried out at decreasing pressure levels, and the viscosities are computed for each

TOP VIEW OF FLOAT

GAS INLET
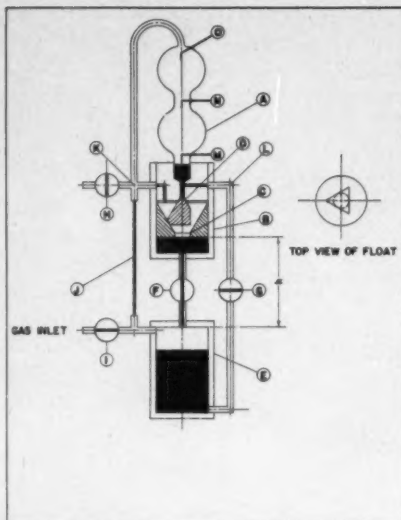
Schematic diagram of the new flowmeter which the U.S. National Bureau of Standards has developed for measuring the rate of flow of very small volumes of gas through capillary tubes. The capillary tube (J) is connected into the meter to form a closed system in which the gas under study flows out of a reservoir (E) and through the capillary tube to displace mercury in the calibrated volume chamber (A). The float valve (D) regulates the flow of mercury out of the volume chamber as it is displaced by the incoming gas and also maintains constant the height (h) of the mercury column which supplies the hydrostatic pressure causing the gas to flow through the capillary tube. As the displaced mercury passes out of the calibrated volume chamber, it breaks contact successively with the electrodes (O), (N) and (M), thus activating a timing circuit. The pressure in the float-valve chamber is made equal to that in the calibrated volume chamber by the connecting tube (K). The valve (H) connects the apparatus to a vacuum system for preliminary purging of gas. The gas sample to be studied is admitted to the flowmeter through the valve (I). The tube (L) and valves (F) and (G) are used in filling the reservoir (E) with gas and the volume chamber with mercury before measurements are begun.

value of pressure. This procedure gives a family of isotherms showing the variation in the viscosity of the gas sample with pressure at the temperature of the test.

In addition to its use in viscosity studies, the new flowmeter may find application in determining the specific gravity of a gas by the effusion method. For this purpose the capillary tube would be replaced by a thin metal diaphragm containing a small orifice.

Because the viscosity of a gas changes rapidly with temperature, the new flowmeter should also prove useful for measuring temperatures, particularly at elevated levels where thermoelectric means have proved unreliable. For such use, the capillary tube would be coiled into a compact sensing unit which would be charged once and for all with a suitable gas. The time interval for a given volume of the gas to flow through the capillary would then be a measure of the average temperature of the capillary.

Recent publications

Pure CO₂ from oil. The Girdler *Girbotol* process, which extracts CO_2 from the flue gas produced by burning fuel oil or fuel gas, is described in a six-page pamphlet issued by the Power-Gas Corporation Ltd. This plant produces either liquid or gaseous CO_2. It is made to the general designs of the Girdler Co. and is similar to units built and used extensively in the U.S. by army, navy and commercial concerns. The equipment (covered by British Patent 655,401) is capable of sustained production of high purity CO_2. Two standard sizes of plant are manufactured, one designed to produce 150 lb./hr. CO_2 and the other 300 lb. A third size producing 50 lb./hr. CO_2 is also available.

Ball and pebble mills. Steele and Cowlishaw's new catalogue illustrates the diversity of their *Steel-Shaw* ball and pebble mills, which range from the water- and steam-jacketed type to mills with special linings, such as silex, granite, porcelain or steel. A variety of driving gear is illustrated, such as worm reduction gear and belt drives. Dry grinding mills are shown with a wood or metal casing to eliminate the spreading of dust while the material is being discharged. It is pointed out, however, that it is possible to adapt any *Steel-Shaw* mill for dry grinding. Various grinding media, such as porcelain or stearite balls, flint pebbles, hardened steel balls, forged steel or hard white iron balls, are illustrated.

The world of oil. In a booklet with this title the Petroleum Information Bureau attempts to explain the petroleum industry to the man in the street. It also tries to give the very many users of oil products an insight into the long sequence of operations between the discovery and production of crude oil and its later conversion into refined products. The booklet covers the whole process of oil production from the preliminary geological search through the drilling, production, transportation (by pipelines and tankers), refining, to the chemicals derived from petroleum. It is available free from the Bureau.

Centrifugal pumps manufactured in stainless steel and other high-nickel corrosion-resisting alloys are described in a 4-p. illustrated pamphlet from the Howard Pneumatic Engineering Co. Ltd. The pumps are of two classes: those for the chemical industry, which are of the normal type with closed impellers and having flanged-end connections; and those for the food industry, which have all internal parts machined all over and are designed for easy cleaning.

Both types of pumps have circular casings which give a wide range of duties for a given size of pump. All pumps are of the single-stage type with external bearings of ample size. The pumps cover a range of outputs from 5 to 300 gal./min. and heads up to 140 p.s.i.

Mechanical Engineering Research

THERMODYNAMIC MEASUREMENTS AND CENTRIFUGAL PUMPS

LABORATORIES for the study of heat transfer and of high-temperature creep and fatigue are among new buildings planned by the Mechanical Engineering Research Laboratory of the D.S.I.R., whose annual report has just been published.* Work carried out by the laboratory in 1953 that is particularly interesting to chemical engineers include investigations on thermodynamic measurements and on centrifugal pumps. Progress in these fields is described below.

Thermodynamic measurements

The Thermodynamics Committee has from the time of its formation stressed the need for more experimental and theoretical work on the properties of mixtures of gases. The large number of mixtures of practical importance precludes direct measurement for each one, and makes the development of theoretical generalisations particularly desirable.

The thermodynamic properties of a gas of sufficient dilution are almost entirely determined by encounters between two molecules. If, therefore, three pure substances and their binary mixtures are studied, sufficient information about the intermolecular forces for similar and dissimilar pairs of molecules can be obtained to enable the low- and medium-density properties of mixtures of all three substances to be predicted. For certain substances the work of Longuet-Higgins and collaborators has facilitated the calculations; it predicts the behaviour of their mixtures in the two-phase and critical regions with fair accuracy.

To obtain experimental data for testing the theoretical methods which have been developed, three lines of work have been encouraged:

(1) An extra-departmental contract has been made with the Imperial College of Science and Technology, University of London, for work on the latent heats of binary mixtures.

Direct measurements of the integral flash latent heat at constant pressure of the carbon dioxide/ethylene system were published in 1951.† Some limitations of the method then became apparent, and it was decided to approach the problem indirectly by measuring Joule-Thomson coefficients and phase equilibria over a wide range of temperature and pressure. A new expansion valve was developed, particularly to minimise the leakage of heat, and the apparatus is giving satisfactory results.

This extra-department contract ended

*'Mechanical Engineering Research, 1953,' H.M.S.O., 2s. 6d.

†A. J. Barnard, S. P. Luthra, D. M. Newitt and M. U. Pai, 'Two-phase Equilibria in Binary and Ternary Systems,' Proc. roy. Soc., A, 1951, 209, 143.

in September 1953, but the general programme of which the work supported by the laboratory formed a part continues with other financial arrangements.

(2) An extra-departmental contract has been made with Manchester University to determine isothermal enthalpy-pressure coefficients, pressure-volume-temperature-composition data and other relevant quantities. This contract has also had the effect of encouraging theoretical work in the University which, while not part of the contract, bears strongly on the whole subject of the thermodynamic properties of mixtures.

Measurements of isothermal Joule-Thomson coefficients of mixtures have been made in the temperature range 0 to 45°C. for the mixtures carbon dioxide/nitrous oxide, carbon dioxide/ethylene, and nitrogen/nitrous oxide. The widest possible temperature range is desirable to determine the constants of the intermolecular forces. New apparatus has been designed and constructed to extend the temperature range to 100°C. and above.

A dew- and boiling-point apparatus, developed by Cook for pressure-volume-temperature composition measurements, has been refined for greater accuracy and results for the carbon dioxide/nitrous oxide system have been published. The agreement with theory is encouraging. An approximate method of deriving the virial coefficients of mixtures has been checked, using the University's electronic computer, with satisfactory agreement.

The principle of corresponding states has been used to compare the viscosity and diffusion coefficients of gas mixtures with those of pure gases. This work, which has been published, has provided information about the inter-molecular potentials which agrees with that derived from the Joule-Thomson coefficients and from the critical constants. The theory of regular solutions is being further examined to establish the conditions under which it can be regarded as valid. A theoretical paper on deviations from the law of corresponding states for elliptical and dipolar molecules has been published.

(3) Work is also being carried out at the National Physical Laboratory on the measurement of pressure-volume-temperature properties of pure substances and binary mixtures. A special study is being made of carbon dioxide, propylene and ethylene. This work is not part of the programme of the Mechanical Engineering Research Board, but forms an integral part of a general attack on the problem of mixtures.

Centrifugal pumps

For many years centrifugal pumps have been used in a wide range of fluid-transport problems and much effort has been devoted

to improving their performance. Their design, however, rests more on practical experience than on basic knowledge; in spite of marked improvements in centrifugal compressors for aircraft engines achieved under the impetus of war, little of the information is available in a sufficiently generalised form for use by pump designers.

More knowledge is required of the behaviour of an impeller when running in a casing. The problem of the conditions which affect the efficient transfer of energy of the fluid from impeller to casing has been the subject of several classic researches. In the experiments being carried out at East Kilbride, attention is particularly directed to conditions in the volute and the effects of impeller/volute interaction.

Research is being undertaken on a proprietary pump of about 400-gal./min. capacity and precision measuring instruments have been developed to facilitate the work. The 25-h.p. test rig, which is of novel design, provides special means for controlling and measuring speed and was described in the report of the laboratory for 1952.

The readings of pressure and direction of flow under specified conditions of pump operation are remarkably steady and consistent; over a period of some weeks the pressure variation was less than $\pm 0.1\%$ and the yaw variation less than $\pm \frac{1}{4}°$. The programme of work includes dynamic measurements of flow and pressure using electrical pickups, sensitive to high frequencies, inserted directly into the volute.

The results so far obtained show that substantial energy losses can occur between the impeller outlet and the discharge flange and that these losses increase rapidly as the flow increases beyond that at the point of maximum efficiency. This effect has been correlated with variations in pressure and flow occurring round the volute. The geometry of the volute at and near the 'cutwater' has been found to be a critical factor.

Thermo-electric pyrometers. Foster Instrument Co.'s new 70-page catalogue No. 112 supersedes all previous issues. There are sections on indicating and recording instruments, instrument panels and portable potentiometers. There are also several sections on thermo-couples, sheaths and accessories. Brief technical notes on the theory and development of the thermo-electric type of pyrometer are given. The range of equipment covers the entire field of temperature measurement from sub-zero to the highest encountered in industry or research. There is a full index and a questionnaire for use when ordering a pyrometer.

Plant and Equipment

Improved viscometers

Three types of viscometers were displayed by Ferranti Ltd. at the recent Physical Society Exhibition in London. The design features of the new improved Ferranti-Shirley cone and plate model include constant shear rate and shear stress throughout the fluid sample, which may have a volume of less than 0.1 cc. and a mean layer thickness of 0.001 in. The rate of shear is continuously variable over the range 0 to 25,000 sec.' and stress-induced heat at high shear rates is rapidly dissipated, greatly reducing the temperature rise in the fluid. Filling and cleaning operations are reduced to the lowest practicable time, it is claimed.

Portable viscometers with extended cylinders for measurements in deep tanks or low liquid levels and thermally insulated cylinders for high-temperature operation were exhibited. Both types of cylinder may be fitted to the standard viscometer.

A modified version of the portable viscometer was also shown which allows a continuous flow of liquid through the measuring annulus. A d.c. signal proportional to the viscosity is provided, capable of actuating standard recording and control gear. Features of the instrument include the ability to match the shear rate conditions obtaining in a process and zero step-up compensation giving expanded scale operation.

Diaphragm compressor

A new diaphragm compressor is claimed to compress all known gases easily, economically and without danger. It is suitable for refrigeration installations where ammonia, carbonic acid gas or freon is used, as well as for applications in the chemical and other industries. The makers of this equipment, the French firm of Corblin, Paris, describe it as follows:

The Corblin diaphragm compressor is essentially made up of two cylindrical plates with conically ground-out bearing surfaces held together by bolts which clamp a flexible metal diaphragm.

One of the plates is perforated with holes through which the double-cone chamber communicates with a pump; the other plate bears the valve boxes for gas suction and discharge.

A piston acts on the oil with which the pump barrel is filled, thus giving the diaphragm its oscillating motion and hence gas suction and delivery.

A small compensation pump replaces the oil that escapes between cylinder and piston at each stroke and applies the diaphragm against the plate which carries the valve boxes, thus reducing the detrimental gap to a minimum. The excess oil is driven out at the end of the stroke by
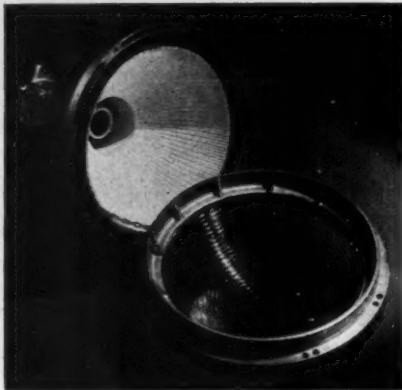
One of the new range of weatherproof and flameproof lighting fittings.

a pressure reducer made of a spring-adjustment valve and the oil returns to the casing.

Since the piston works in oil and since the diaphragm separates the gas from the oil completely, the compressor operates without stuffing box; this, added to maximum volume output ensured by the small compensation pump, allows the compressor to work with the minimum amount of motive power.

The gas to be delivered comes in contact only with the metal diaphragm; hence it is compressed in absolutely pure state without trace of lubricant and, on the other hand, no gas can leak outside.

The diaphragm is not mechanically connected with the piston; it moves under the impulse of the oil which follows the motions of the piston. This piston is equipped with metal rings, not cup leather or other packing. Bearings and crank heads are equipped with ball- or roller-bearings. Three screws bearing against the lower plate are used to raise the upper plate to free the diaphragm, which can thus be easily replaced. The slight lift ring, or disc valves, are readily accessible.

Weatherproof and flameproof lighting fittings

A new range of weatherproof/flameproof lighting fittings for use in exposed positions are now being produced by Victor Products Ltd.

This new range is for 100 to 150, 150 to 200 and 300 watts for Groups I, II and III. All three fittings comply with B.S.S. 889, and it is claimed that complete weatherproofing is achieved by the incorporation of gaskets on all joints. Other features included top or through-way junction box for conduit, *Pyrotenax* or cable; internal reflector; shallow diffusing glass; anchored by chain; suspension lugs and smooth lampbody lines.

Measuring the quality of carburising and carbonitriding atmosphere

A new instrument for measuring the quality of a carburising atmosphere consists essentially of a compartment (with a transparent front) inside which is an iron wire which is carburised in the atmosphere under test and cooled rapidly so that it is transformed to martensite. In this condition the electrical resistance of the wire is a measure of its carbon content and hence of the carburising potential of the atmosphere. Values of the electrical resistance of the quenched wire can be directly related to carburising potential from a calibration curve. Operation of the gauge is straightforward and one measurement takes about 15 min.

The carburising potential measured on the carbon gauge is the carbon content of a steel with which the atmosphere would be in equilibrium at a given temperature. For example, if the carburising potential of an atmosphere is measured on the gauge as 0.65 C, then this atmosphere would be neutral to a 0.65 C steel at the same temperature. This method of evaluating the quality of a controlled atmosphere is particularly useful in such processes as the hardening of medium or high carbon steel where decarburisation is to be avoided, the restoration of carbon to surfaces of parts which have been decarburised during casting or heat-treatment and the carburisation of steel to some specific value of carbon content. Makers are the General Electric Co. Ltd.

Mobile radiographic unit

Engineering radiography is not confined to the examination of constructional welds for certification purposes. It can be used to check sample welds during the selection of welders in the field. In addition, as a method of quality control, periodic spot-check photography enables a consistently good standard of welding to be reached and maintained on ordinary commercial-grade work in the field.

There are many engineering firms which would find radiography an invaluable aid for the progressive non-destructive testing of welding quality during the construction of chemical pipework, storage tanks, gasholders, pressure vessels, blast furnaces, metal structures, castings, etc., but to whom the maintenance of a permanent radiographic unit would not be an economic proposition. For such firms, the Power-Gas Corporation Ltd. and Ashmore, Benson, Pease & Co. have made available a mobile radiographic unit. Carrying its own water storage tank and electric generating set and storage battery, the unit is quite independent of site water supplies and, if gamma-ray isotopes are used, of site power.

The largest item of the radiographic equipment is mounted on a Ford 8 chassis and is loaded, *via* a ramp, into the rear part of the vehicle. This compartment is fitted

out for use as a viewing room and site office after the x-ray and gamma-ray equipment have been unloaded. The unit also includes an air-conditioned dark room provided with hot and cold running water. Within a short time of the radiographic examination being completed the film can be processed in the dark room, dried and examined. The radiographs provide a permanent record of the quality of the work examined.

The radioactive isotopes in their protective lead- and tungsten-lined containers are stored in two cavities under the floor of the unit. Special precautions are taken to safeguard the health of people working in the vicinity of radioactive materials.

The equipment and methods used vary according to the type of work under examination. Depending on the gauge and size of the work, the materials involved—whether steel, nickel, aluminium, or light or heavy alloys—and method of production, *e.g.* castings, choice is made of x-ray photographic units ranging from 150 kv. to 300 kv. or gamma-ray photography using one of the radioactive isotopes Iridium 192, Thulium 170 or Cobalt 60.

New flotation mechanism

A new suspended unit shaft assembly for their ' Sub-A' flotation machine, designed to reduce maintenance time, is announced by Denver Equipment Co., manufacturers of mining equipment. Assembled as one unit, the shaft assembly is suspended in the tank by four bolts. Removal of the four bolts allows the mechanism to be lifted from the cell (see illustration). Removal and replacement can be made in less than 10 min. Parts for the new shaft assembly are interchangeable with parts for older models. A removable feed pipe enables the assembly to be adapted for either standard cell-to-cell or free flow of pulp operation.

Automatic acid-handling system

An automatic acid-handling system installed in the paper-making plant of Robert Gair Co., New York, has, it is claimed: (1) eliminated dangers of using sulphuric acid at the wet ends of paper machines; (2) provided an accurate method of controlling paper stock acidity; (3) saved storage space; (4) improved personnel safety; and (5) reduced costs.

The new system incorporates a 1,500-gal. storage tank located in a remote corner of the plant's basement, an acid pump, a control panel and a mixing tank. The storage tank is filled directly from a tank truck outside the plant. The mixing tank is automatically filled by the pump. The control panel has two indicators on it. One shows the dilute acid level in the mixing tank; the other shows the concentration percentage.

When the mixing tank level falls below a predetermined point, the level indicator opens a switch that admits water into the
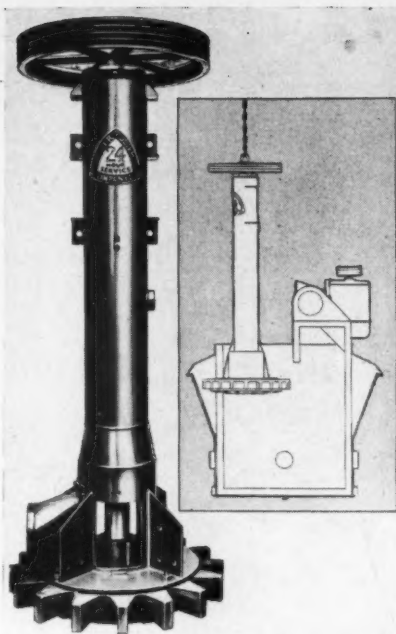
Suspended unit shaft assembly for flotation machine. It is suspended in the tank by four bolts and can be removed and replaced in 10 min., it is claimed.

tank. As water is added, the acid concentration change is pneumatically transmitted to the other indicator, which closes a contact and starts the acid pump. These automatic controls regulate the amount of sulphuric acid which flows from the mixing tank to the screens on both machines to provide an accurate control of stock acidity. When the proper tank level is reached, the water valve closes. When the dilute acid reaches the proper concentration, the acid pump shuts off.

This method of handling acid in a well-arranged system not only provides a more economical means of increasing paper output but is an improvement over the plant's previous machinery layout. The old arrangement, which used a mixing tank on the second floor, caused the screen man to waste time and labour in moving carboys of acid from storage and emptying them into the tank. This not only required more space but created hazards to personnel.

To sum up, the system improves personnel safety, saves time and storage space, and saves money by permitting acid to be purchased in bulk quantities.

For further information about the Plant and Equipment described, use the coupon on page 334

Safety testing of electric tools

The *Terra-Tertia* test unit type 121 is a safety device for rapidly testing the earth conductor efficiency of portable electric tools. Thus, storekeepers can check the earthing systems of all portable electric tools before issue and in this way faulty tools can be set aside for servicing before serious injury or damage can occur. Again, semi-skilled workmen can ascertain for themselves, at any time, that their appliance, associate lead and plug are in a thoroughly serviceable condition.

The test unit is connected to a.c. mains supply, 220 to 250 v., 50 c/s, and the tool to be tested is plugged into the 15-amp. three-pin socket on the front panel, and with the body of the tool pressed against the instrument's handle the panel switch is then depressed. In this arrangement a high current at low voltage flows through the earth conductor and associate plug, which, if satisfactory, withstands the current flow and lights the ' Pass ' lamp on the front panel. If the earthing system is faulty, then the lamp will not light. Therefore, should the lamp itself fail, it is impossible for the tool, faulty or otherwise, to be passed as safe.

A frequent danger in portable electric tools is the partial fracture of the earth conductor due to negligence or wear and tear. In such cases the instrument's high test current at low voltage is sufficient to completely burn out the remaining strands of the partially fractured earth wire, thereby anticipating the failure during work.

The unit, which is made by Electro Methods Ltd., is contained in a robust case with steel carrying handle. Its weight is $12\frac{1}{2}$ lb.; its dimensions are 8 in. by 7 in. by $6\frac{1}{2}$ in.

Fire pump powered by gas turbine

For many years Sigmund Pumps Ltd. have attempted to design a fire pump which can give the fire fighter the essential qualities demanded by the urgency of the job—light weight, small bulk, easy and quick starting, fast priming and absolute dependability at all times.

Now, advances in gas turbine design and development by the Rover Co. have been brought together with Sigmund hydraulic experience to create a new fire pump. It combines the advantages of the latest Rover lightweight gas turbine (see accompanying note) with those of the Sigmund high-speed centrifugal pump and gives, it is claimed, advantages not obtainable previously by equivalent portable petrol-driven fire pumps.

Its features include: light weight and small bulk, easy and quick starting, easy and quick priming, can be run on diesel oil, paraffin or petrol fuel, and standard N.F.S. fitting—twin discharge branches, grouped starting and priming controls, grouped instrument control panel and frame of braced tubular steel construction, giving maximum strength with lightness.

Chemical Engineering Invention

RECENT BRITISH PATENT CLAIMS

### Extracting potassium from dilute solutions

Potassium is extracted from solutions containing it in small quantities, *e.g.* sea water, etc., by treating the solution with a solid highly nitrated secondary aromatic amine and a base, *e.g.* lime or ammonia, precipitating thereby a slightly soluble potassium salt of the amine, separating it and treating with an acid, recovering thereby the amine in solid state for reuse, and a comparatively strong solution of the potassium salt. A *p*H of 7 to 8 should be maintained during the aminate precipitation. The reaction mixture passes to a thickener, and the clear liquor therefrom is treated with acid for the removal of small quantities of dissolved precipitating agent, followed by filtration in sand filters and if desired a charcoal filter, and deacidification with calcium carbonate.

A small quantity of acid is added to the material from the thickener to reduce the *p*H to about 3, and the fine amine crystals produced and the fine aminate crystals, say, 50% by weight of the total, are returned to the precipitating tank. The coarse crystals are treated with acid, *e.g.* nitric acid, and the amine precipitated is finally suspended in, say, sea water for reuse. The bicarbonate content of the sea water may be reduced prior to treatment. Amines specified are dipicryl amine, and various highly nitrated chloro-, methyl-, methoxy-, and ethoxy-diphenyl amines, Specification 605,694 is referred to.— 668,735, *Aktieselskab Norduco.*

Cyclic re-forming of natural gas, etc.

A cyclic reforming process for the production of a gas rich in hydrogen and carbon monoxide from a hydrocarbon gas such as natural gas comprises, in one part of the cycle, passing hot combustion gases serially through a heat storage bed and a catalyst bed and, in another part, passing natural gas with steam through the heat storage bed wherein the mixed gases are preheated, and then through the catalyst bed wherein the reforming of the gases occurs.

The process is carried out in apparatus consisting of a refractory lined shell 1 provided with two arches 16 and 17, the lower arch supporting a bed 15 of refractory material and the upper a mass 4 of catalytic material, *e.g.*, metallic nickel deposited on alumina. The process is started by admitting oil or gas through line 5 and air through line 6 so that the fuel burns, the hot gases heating the beds 4 and 15 to about 1,600°F. and escaping through stack 7. After a steam purge, hydrocarbon gas and steam are admitted through lines 8 and 9, air also entering through line 10 to maintain the operating temperature.
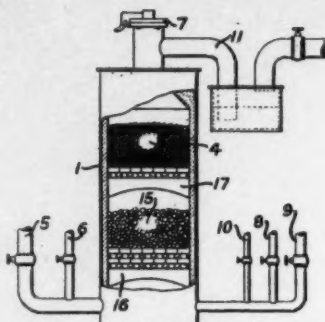
The gases are preheated in bed 15 and react in bed 4 to give a gas rich in hydrogen and carbon monoxide, which discharges through line 11. In an example, natural gas was reformed to give a gas containing 40.7% hydrogen and 12% carbon monoxide.—668,978, *United Engineers & Constructors Inc.*

Extracting fish-liver oil

Fish livers are mechanically grated, mixed with hot water, and centrifuged in a separator provided with outlet nozzles in the bowl wall through which water and solid particles are discharged as a sludge. A part of the sludge is reintroduced into the sludge space of the bowl in order to minimise the quantity of hot water used, especially in the case of apparatus installed on motor trawlers.
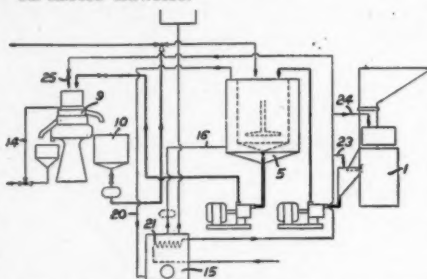
In the installation shown, the livers are introduced into a liver grater 1, preferably of the type described in Specification 614,972, and pumped into a stirred, double-jacketed heating tank 5 and then to a centrifuge 9 of the type referred to above. The oil is collected in a tank 10 and pumped either direct to storage or through a heater and cylinder separator, when a further quantity of sludge is separated. The sludge concentrate discharging from the nozzles in the bowl is partly reconducted to the sludge space of the bowl through the pipe 14.

Water for mixing with the liver mass, the grated livers, and the material in the centrifuge, is heated by a coil 21 in an oil-fired boiler 15 and delivered through pipes 24, 23 and 25, while hot water for circulating in the jacket of the tank 5 passes directly from the boiler through the pipes 16 and 20. Ordinary sea water may be used if desired.—658,261, *Aktiebolaget Separator.*

Production of cyclised synthetic rubber

Aqueous dispersions of cyclised synthetic rubber are obtained by reacting a cyclising agent, *e.g.* hydrofluoric acid and sulphuric acid, with an acid-resistant aqueous synthetic rubber dispersion or a flocculate. Heat and pressure may be applied. Synthetic rubbers specified are polyisoprene, polydimethylbutadiene, polymethylpentadiene and their copolymers. The latex may be rendered resistant to coagulation with acid by adding casein, saponin, or haemoglobin, but is preferably stabilised by adding cetylpyridinium bromide, dialkylaminoethyloleylmethosulphate, *Igepon T*, dodecylphenylpolyglycol ether, or polyglycol ether.

The cyclised latex may be modified by adding plasticisers, *e.g.* dibutyl phthalate, stabilisers and fillers and the cyclised latex or the cyclised rubber resulting therefrom may be vulcanised with S, sulphides, S_2Cl_2, $ClSO_3K$ or SO_2Cl_2. Specification 634,879, [Group V], is referred to.—658,520, *Rubber-Stichting.*

Triple superphosphate manufacture

Triple superphosphate is produced by intimately mixing ground phosphate rock with phosphoric acid, preferably of 34 to 40% strength, until a slurry is formed, which is passed to a slowly moving inclined surface whose speed of movement is so adjusted that the slurry, retained thereon until after passing through a porous mass phase, is disrupted into particulate form by chemical and physical action. The slurry may thus be delivered to an inclined endless belt conveyer, the inclination of which is adjustable, the granules from the discharge end passing to a dryer in which they are hardened.

Alternatively, the slurry is fed to a slowly revolving rotary conditioner whose wall constitutes the slowly moving inclined surface, and thence to a second faster moving rotary conditioner wherein granulation of the disrupted product is completed. Fines from the usual screen apparatus may be returned to the discharge end of the mixer.

The ground phosphate rock employed may be subjected to differential air separation, the finer fraction passing to the reaction mixer and the coarser fraction to the phosphoric acid production plant as described in Specification 620,590. Specifications 503,352, 559,482, 596,782, 625,863, and U.S. Specification 2,070,582 are also referred to.—668,269, *Sturtevant Engineering Co. Ltd., H. Richardson & Co. (York) Ltd., J. T. Procter and A. Ogilvie.*

Stress-Relieving a Large Welded Pressure Vessel

AN interesting problem which was encountered recently consisted of stress-relieving the central weld of a high-pressure oil fractionating column, 120 ft. in length and 3 ft. 8 in. in diameter. As reported in a note from the East Midlands Gas Board, the column had been initially fabricated in two 60-ft. sections, each section being separately stress-relieved in a furnace. The two sections were then aligned for the final central weld at the works of a local chemical engineering firm. As there was no available furnace large enough to take the whole of the completed column, it was agreed that, if this final weld was stress-relieved on site to a predetermined heat-treatment specification, the column would be accepted by the oil company's inspecting engineer. This called for a temporary furnace setting local to the weld.

Conditions required for heat treatment

These were taken from a Code of Practice issued jointly by the American Petroleum Institute and the American Society of Mechanical Engineers in 1943, relating to the heat treatment of pressure vessels, an abstract from which is given below:

(a) The temperature gradient on either side of the furnace and along the heated portion of the shell not to exceed 200°F./ft.

(b) The temperature difference throughout the portion of the shell being heated not to exceed 150°F.

(c) The rate of heating above 600°F. not to exceed 400°F./hr.

(d) A temperature of 1,100°F. to be attained and maintained for 1 hr.

(e) The rate of cooling not to exceed 500°F./hr.

The furnace

The process for which the column was designed necessitated careful heat treatment, calling for an even temperature around the welded section and a close control of that temperature. A furnace was therefore built to the design shown in Figs. 1 and 2.

A brickwork duct was built around the lower half of the vessel, the construction being such that a rectangular combustion chamber was formed with an offtake into the duct, and a flue arranged at low level to relieve any excess pressure in the furnace.

The upper half consisted of a sheet-steel duct which rested on, and was sealed to, the lower brickwork. This duct was then covered with brickwork to form the finished structure as shown in Fig. 1.

With a view to maintaining the required temperature gradient along the column, a ring of low-temperature insulating bricks 3-in. thick was placed against the interior surface for a total length of about 6 ft., and a spillage of hot gases from the heating chamber was allowed to flow along the
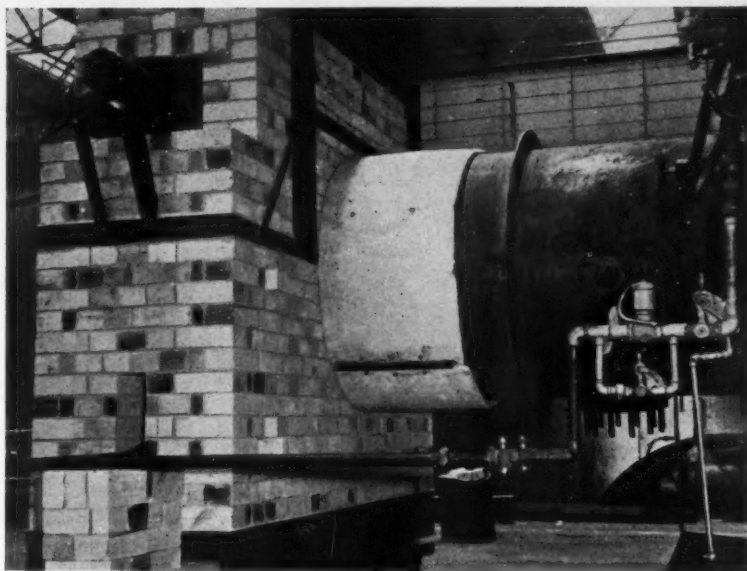

Fig. 1.

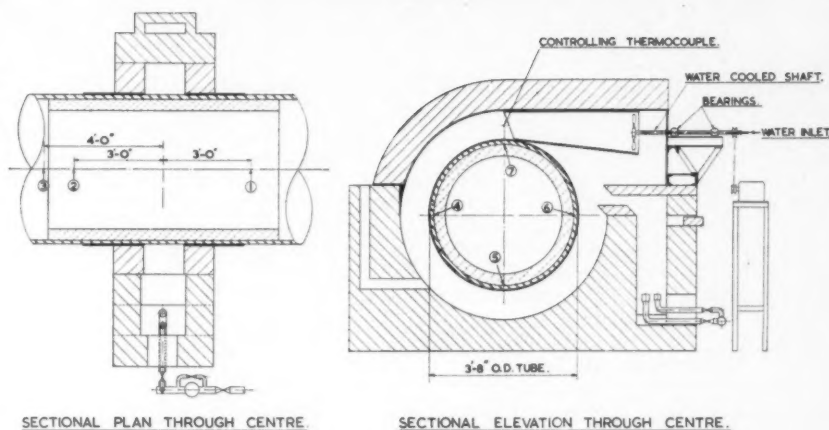SECTIONAL PLAN THROUGH CENTRE.   SECTIONAL ELEVATION THROUGH CENTRE.

Fig. 2.

exterior surface of the vessel for a distance of some 2 ft. on each side of the main chamber in an annulus created by the fitting of lagged sheet-steel covers held away from the shell by ¼-in. distance pieces.

The portion to be heated was a circumferential strip 15-in. wide, and in the method adopted the hot gases were drawn from the top of the combustion chamber into the inlet side of a high-speed axial-type fan and circulated at high velocity around the annular space between the exposed shell surface and the ducting. It was essential in this design to have the fan blades and a portion of the shaft in the flow of the hot-gas stream. In order to avoid any possibility of overheating the independent fractional-horse-power motor, it was proposed to employ a vee-rope drive and to water cool the hollow fan shaft and the shaft bearings. In practice, however,

the fan shaft was solid and driven direct by a motor through insulated couplings. This was not entirely satisfactory, as the conducted heat affected both the bearings and the motor during the course of the tests.

Burner equipment and temperature control

The burner equipment consisted of two *Hypact* burners, Nos. 600 and 400, each separately controlled by a quadrant cock and fed from a common-gas-supply pipe which carried the control equipment. The gas was fed to the burners through a a constant-pressure governor and a solenoid valve, the latter being by-passed with a small valve in the by-pass pipe for adjustment purposes (Figs. 1 and 2). The solenoid valve was operated from the electricity mains through a temperature indicator/controller, and the controlling

thermocouple was sited in the hot-gas stream at its first point of contact with the vessel.

In order to assess the average temperature of the heated section, it was felt necessary to obtain further temperature recordings both around the weld and along the vessel away from the weld. For this purpose four thermocouples (Nos. 4, 5, 6 and 7, shown in Fig. 2) were sited on the inner surface of the vessel around the weld. A further three thermocouples (Nos. 1, 2 and 3, also shown in Fig. 2) were sited at appropriate points along the inner surface of the vessel away from the weld. In order to determine the rate of temperature rise, No. 7 ' couple ' was connected to a temperature recorder, and half-hourly readings of the remaining ' couples ' were taken by means of a potentiometer, which was also used to check the recorder.

Performance

As rapid heating was not essential, it was decided to avoid any possible local strains due to expansion by bringing up the temperature slowly. This was done by running the No. 400 burner until the temperature rise commenced to slow down, then changing over to the No. 600 burner, and finally running both burners full-on against control by operation of the solenoid valve from the temperature controller. The whole cycle was carried out to the satisfaction of the inspecting engineer and the completed column was accepted by him.

The gas consumption over the total heating cycle was approximately 20,000 cu. ft. (100 therms). This was considered to be reasonable in view of the size of the vessel and the nature of the heat treatment involved.—(*Information supplied by the East Midlands Gas Board.*)

interdiffusion of titanium and brazing alloy. Methods of improving joint properties are discussed. Better torch-brazed joints might be obtained through improved techniques, alloys and fluxes. Metals which should prove useful as a barrier layer are suggested. Other conclusions are drawn regarding optimum temperature and joint thickness.

In a report on the brazing and soldering of titanium and its alloys by the Armour Research Station, Illinois Institute of Technology, it is stated that silver, copper, manganese and tin-based fluxes were found suitable for brazing and soldering commercially pure titanium. Brazing methods involving heating by oxyacetylene torch, by means of electrical resistance and in an inert atmosphere furnace were studied. Joints brazed with fine silver break with a ductile shear fracture at approximately 30,000 p.s.i. shear strength. A tentative titanium-silver binary phase diagram showed one intermetallic compound TiAg, unique in that it is highly ductile. This accounts for the ductility of the brazed joints. Most other braze metals form brittle intermetallic compounds with titanium. Recrystallisation bonding of titanium has produced high - strength lap joints. Methods for soldering titanium with a soldering iron were also developed.

Steam Raising at Britain's First Atomic Power Station

JUDGING by the perspective drawings that have been made of the Calder Hall, Cumberland, atomic power station, the layout comprises a central turbine house, with a building at either end containing a nuclear reactor. Symmetrically disposed about each reactor building are four steam-raising towers. A noticeable feature is the absence of conventional chimneys, since no oil or coal will be burnt. Instead, two comparatively small stacks are provided on each reactor house for venting cooling air.

The steam-raising towers, which with their cylindrical steel shells and surrounding catwalks are more reminiscent of an oil refinery than of a power generating station, serve as a reminder that, even in an atomic power station, steam is wanted for motive power. Heat generated in the graphite-moderated uranium reactors is conveyed to the steam plant by carbon dioxide under high pressure; the hot gas gives up its heat to preheating, evaporating and superheating sections of the steam plant. After passing through the steam-raising towers, the cooled gas is recirculated by powerful blowers back to the reactor, where the cycle is repeated.

From Babcock & Wilcox Ltd., at whose Renfrew works the eight 80-ft. steel towers are now under construction, come details of the steam-raising plant. One of the most interesting features is the use of a double-pressure steam cycle in order to obtain maximum efficiency from the comparatively low temperature (as compared with a conventional furnace) of the gas entering the steam towers. Each tower contains, in effect, two separate sets of steam-raising equipment, there being separate H.P. and L.P. economiser, evaporator and superheater sections, each with its own drum and feed pump. The turbine plant will, of course, be designed with a L.P. section which will receive live steam direct, as well as steam which has already been expanded in the H.P. blading.

In order to obtain the maximum heat transfer within the towers all heating surfaces are of stud-tube construction to give extended surface. The studs are flash-welded and have smooth surfaces and a profile giving good aerodynamic properties.

An important requirement of the internal heating surfaces is that there should be no leakage of steam or water into the coolant gas, otherwise contamination of the internals of the reactor—the fuel elements and graphite—would result. The design, therefore, involves no joints inside the pressure shells except those which, once completed in the shops and exhaustively tested, will not be required to be broken throughout the anticipated life of the plant. In addition, the high pressure of the coolant gas (due to the need for economy in blower power) requires the whole system to be built to withstand this pressure without any possibility of leakage.

Babcock's are responsible for the complete steam towers and their associated feed pumps, etc. The reactors are being built by the Department of Atomic Energy, the pressure vessel or outer shell for the reactor being made by Whessoe Ltd. The circulating gas blowers and pipework are by C. and A. Parsons & Co. Ltd., who are also supplying the turbo-alternator plant. The main contractors for the station building are Taylor Woodrow.

Brazing and Soldering Titanium

Titanium can be brazed to titanium, mild steel and stainless steel with several commercial brazing alloys by furnace- and torch-brazing methods. This is stated in a report by Lewis, Rieppel and Voldritch, of the Battelle Memorial Institute, Columbus, Ohio. Joints were brittle except where silver or a silver manganese alloy was used. The shear strengths of the best joints obtained were from 14,000 to 19,000 p.s.i. Brittle joints appeared to result from

Projection Welding of Mild Steel

Projection welding is a method of resistance welding in which current flow and heating during welding are localised by the form of the parts being welded rather than by the electrodes in contact with the work. This is usually accomplished by a projection on one or both of the work-pieces, and it is from this fact that the process derives its name. The projection may be formed by pressing, in the case of sheet metal, by forging or machining from solid pieces, or on the edge of strip by using special shears or punches. The advantages offered by projection welding compared with spot welding are as follows:

(1) Ease of obtaining satisfactory heat balance for welding difficult combinations of dissimilar thickness.

(2) More uniform results in many applications, particularly where welds are required at close pitch.

(3) Several welds can be made simultaneously.

(4) Longer electrode life and lower electrode maintenance.

(5) The process lends itself to the use of assembly fixtures.

(6) Finished appearance is often improved.

(7) Parts may be projection welded that could not be otherwise resistance welded.

The whole subject is described in lucid detail in a pamphlet published by the British Welding Research Association, London. It is called ' Projection Welding of Mild Steel ' and costs 3s. 6d.

World News

GREAT BRITAIN

Process exchange agreement

Costain-John Brown Ltd. and Petrocarbon Developments Ltd. have entered into an arrangement with Friedrich Uhde GmbH., the chemical plant designers and engineers, for the mutual use and exchange of processes and process designs, especially in the field of nitrogen and petrochemicals. The full range of these processes and designs, together with the plants embodied in them, will thus be available throughout the British Commonwealth.

Polystyrene manufacture

The Distillers Co. Ltd. announce that negotiations have recently been concluded with the Dow Chemical Co., of Michigan, U.S.A., to form a British company with the name Distrene Ltd. The objects of the new company, in which Distillers will hold the controlling interest, are to manufacture polystyrene and modified polystyrenes, under the Dow Co.'s patents, at Barry in South Wales.

British Resin Products Ltd., a wholly-owned subsidiary of Distillers, will be appointed sole selling agents for the new company.

The directors of Distrene Ltd. nominated by Distillers are: Major C. J. P. Ball, Mr. H. H. Woolveridge, Mr. P. A. Delafield and Dr. J. J. P. Staudinger; and by Dow: Mr. N. R. Crawford, Dr. William H. Schuette and Mr. A. E. Young.

Polythene film factory opened

In their new factory at Stevenage, Herts —the largest polythene film manufacturing unit in the world outside the U.S.A.— British Visqueen Ltd. are now producing *Visqueen* film. The company's products include *Visqueen* C, a specially surface-treated film which overcomes the problem of print adhesion.

The opening of the factory initiates a new phase in the development of thermoplastic packaging films in the U.K. After five years' development work by Imperial Chemical Industries Ltd. with *Alkathene* film, made on a pilot-plant scale, a market had been created of sufficient size to justify large-scale continuous operations. To meet this situation, just over a year ago I.C.I. Ltd. pooled resources with the Visking Corporation of the U.S.A. to form British Visqueen Ltd. This company's new factory enables large tonnages of *Visqueen* (which has now replaced *Alkathene* film) to be made in a wider range of sizes and at lower cost. In anticipation of production economies, *Visqueen* prices were recently substantially reduced.

British Visqueen will not convert the film into bags, liners, printed reels, etc., but will supply it to the package manufacturing industry.

British plant manufacturers' Canadian enterprise

Mr. H. D. MacMurray, of George Scott & Son (London) Ltd., has returned to England after two years' residence in Canada, where he has been representing his firm's interests since the formation of a Canadian subsidiary company with W. S. Barron & Son Ltd., of Gloucester.

This subsidiary company, working in conjunction with Dominion Wheel & Foundries, of Toronto, on the design and manufacture in Canada of the plant of the two British companies, is now making satisfactory progress in what Mr. MacMurray reports as an attractive but by no means easy market.

They are manufacturing some equipment in Canada, but the greater volume has been exported from this country, amounting in the past 18 months to over 10 times the initial dollar capital outlay.

Mr. W. H. Moreland, on behalf of the Barron company, remains in Canada for a few more months.

The parent company of George Scott & Son (London) Ltd. is Henry Balfour & Co. Ltd., engineers, of Leven.

British Oxygen awards research scholarships

The British Oxygen Co. Ltd. announces the awards of the following three scholarships: £500 a year, tenable for two years, at London University for research into the nature of rare gases to Mr. B. F. Figgins; £425 a year, tenable for two years, at Cambridge University for research in the Department of Metallurgy to Mr. D. H. Kirkwood; and £425 a year, tenable for two years, at Oxford University for research into the properties of materials at low temperatures to Mr. R. D. McCammon.

Gas pipeline contract

A contract valued at about half a million pounds has been awarded to Costain-John Brown Ltd. by the Wales Gas Board for the laying of gas pipelines in North and South Wales. These pipelines are scheduled for completion in 1956.

Change of address

Owing to expansion of business in the north and north-west of England, the Manchester and North Midlands branch office of the John Thompson Group of companies is moving to larger premises. The new address is Sunlight House, Quay Street, Manchester 3 (telephone: Deansgate 6151-2).

Change of telephone number

The telephone number of the head sales office of Hess Products Ltd., Leeds, has been changed to Leeds 30194-5.

Improved process gives cheaper polythene

The home trade price of *Alkathene* brand of polythene was reduced by 3d. per lb. to 3s. 2d. per lb. as from September 1. This will make it one of the world's cheapest thermoplastics.

This latest reduction is in line with the policy I.C.I. has followed ever since *Alkathene* was introduced in 1939. Whenever possible, the price has been reduced in order to stimulate demand. These reductions have been made possible by passing on to customers the advantages of I.C.I.'s technical progress. As the result of continued research and development, the economics of the production process have been improved, so that it is now possible to make 30 times as much polythene in unit volume of the expensive high-pressure reaction space as was possible 15 years ago, There is no need to stress the effect of this on cost of production. The oil-cracking process by which the ethylene is made gives a product of the necessary high purity and is the cheapest route available. I.C.I. believes that with continued growth in the scale of manufacture and attention to the detailed economics and technique of the whole process from oil to polythene, *Alkathene* will become as cheap as any polythene in the world.

I.C.I. has also directed very considerable effort towards a better scientific understanding of those factors which affect the consistency and quality of the product, and towards the exploitation of this knowledge to practical ends.

The simple picture of the structure of polythene which was sufficient in 1939 to explain its then known behaviour has now been enormously elaborated, and it is realised that there are a great number of variables which determine the finer details of product behaviour. No single property, such as melt viscosity (grade number), is adequate to describe a particular product, and it is now seen that a whole range of materials having different sets of properties can be made by the polymerisation of ethylene. This is shown up by the slight differences between the several competitive products, some of which are preferred to others for particular purposes. Rate of extrusion, surface finish, toughness and tear strength, low temperature resistance, freedom from solvent cracking and chemical inertness are all of great importance in one application or another, but no one polythene yet combines all the best qualities, and it may be that some properties are mutually exclusive. The situation is such that one polythene cannot be said to be better than another without very strict qualification regarding the use to which it is to be put.

It is I.C.I.'s intention to develop products suited to particular major uses, even if, as is expected, this involves making a range of products. The design of the company's large-scale production units makes it possible to vary synthesis con-

ditions over a very wide range, and it has already been demonstrated experimentally that the I.C.I. process is flexible enough to enable the company to offer polythenes covering the spectrum of available properties. Thus, as existing applications expand and new ones develop, I.C.I. will be in a position to satisfy demand in respect of both quantity and quality.

Contract for Burmese pharmaceutical project

A contract, worth approximately £2 million, has been initialled between the Government of the Union of Burma and Holland & Hannen and Cubitts Ltd., of London, for the civil engineering work, erection of buildings and the necessary mechanical and electrical installations for the Burma pharmaceutical industry. The contract includes provision for a railway line and jetty together with housing for the staff.

The factory buildings will cover well over a quarter of a million square feet of floor space.

Contracts for some of the plant have already been placed by Evans Medical with British firms and more contracts will be placed in the near future.

The architects for the project are James Cubitt & Partners of London.

A mission, it will be recalled, was sent by the Government of Burma to the U.K. nearly a year ago and on October 20, 1953, the agreement was signed between the Government of Burma and Evans Medical for the purpose of setting up a pharmaceutical industry in Burma. Subsequently, James Cubitt & Partners were instructed to draw up plans for the various and considerable buildings required for an industry entirely new to this young country. The speed with which the plans have been drawn up enabled the foundation stone to be laid very much sooner than anticipated.

The similarity between the names of the architects and contractors is coincidental.

The British firm of A.P.V. Co. Ltd. are responsible for supplying the alcohol and yeast plant, an important part of this project.

New agents for Quickfit & Quartz

Quickfit & Quartz Ltd., manufacturers of interchangeable laboratory and scientific glassware, have appointed selling agents in Dublin and in Wells, Somerset.

The Dublin agent is Thomas H. Mason, of 5-6 Dane Street, Dublin, and the agents in Wells are Messrs. Sutherland and Thomson.

The company have also announced the appointment of the Wakefield Co., of Sherbrooke Street West, Montreal 2, as their agents in Montreal and Ottawa.

Tyneside refresher course

A comprehensive refresher course for senior works and plant engineers on Tyneside has been organised by the Incorporated Plant Engineers. It will comprise 19 weekly lectures commencing October 19 at King's

Death of Editor of 'Food Manufacture'

Mr. Thomas Crosbie-Walsh, Editor of FOOD MANUFACTURE and a director of the publishers, Leonard Hill Limited, died on September 7 after a short illness. He was 74.

Mr. Crosbie-Walsh's life was divided between two main interests: chemistry and journalism.
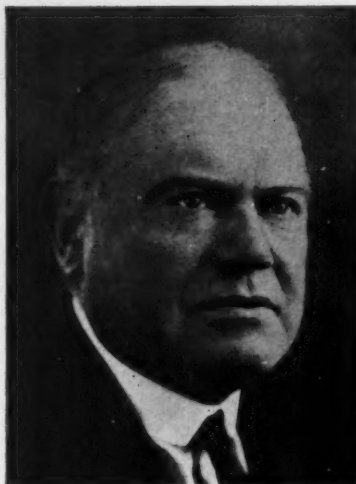
In 1902 he went to South America on behalf of Borax Consolidated Ltd., as chemist, after which he practised as an analyst and consultant in Buenos Aires. In 1914 he was appointed chief chemist of Liebig's Extract of Meat Co. Ltd. He became an Associate of the Royal Institute of Chemistry in 1918 and a Fellow in 1923. After leaving Liebig's he joined Bovril Ltd., and for upwards of two years carried out intensive research work for them.

Mr. Crosbie-Walsh then went to New Zealand to do research and established himself permanently in London in 1932 as a consulting food technologist.

His full-time activities in technical publishing began in 1937 when he joined Leonard Hill Limited as Editor of FOOD MANUFACTURE. During his career with this company he was, in addition, Editor of FOOD INDUSTRIES MANUAL, now in its 17th edition, and, for a time, Editor of DAIRYMAN. He was elected to the Board of the company in 1949.

In an appreciation of Mr. Crosbie-Walsh in the current issue of FOOD MANUFACTURE, Mr. W. Leonard Hill, chairman of the company, writes: 'All his work was characterised by that touch of genius which has been described as " an infinite capacity for taking pains. . . ." To any in the food industry it can be said: If you seek his monument, look around you in your libraries.'

College (University of Durham), Newcastle-upon-Tyne. Full particulars can be obtained from the secretary to the refresher course, Stephenson Building, Claremont Road, Newcastle-upon-Tyne 2.

New plant makes oil refinery self-sufficient in H_2SO_4

The latest addition to Stanlow Oil Refinery, Cheshire, is a plant capable of making 33,000 tons of sulphuric acid a year, which cost £200,000 to erect.

With this plant, which has just gone into operation, and the two-year-old sulphur recovery plant, Stanlow becomes the only refinery in the United Kingdom which is entirely self-sufficient for supplies of sulphur and sulphuric acid. All the acid produced will be used in the refinery for treating and improving lubricating and white oils and in the manufacture of detergents and petroleum-based chemical derivatives.

Sulphur is a constituent of almost all crude oils and must be largely removed from its products before they can be marketed. Some sulphur recovery was carried out in the U.S.A. on a small scale before the war, but it was the post-war shortage of natural sulphur that gave an impetus to the development and installation of these processes in oil refineries throughout the world. In the U.K. there was the added stimulus of the need for dollar-saving.

In 1952, Shell built a plant at Stanlow to recovery sulphur from hydrogen sulphide, which is one of the by-products of oil refining. This plant is now producing about 15,000 tons of sulphur a year. Until recently, in the interests of the national economy, all of this production went to meet the needs of consumers in Britain. Now, however, with the easing of the sulphur supply position, some of it is to be used for the manufacture of Stanlow's own requirements of sulphuric acid.

The processes involved are as follows. Hydrogen-sulphide produced in the refining processes, together with some elemental sulphur, is burnt to produce sulphur dioxide. This is then passed into the sulphuric acid contact plant, which is of conventional design. The sulphur dioxide stream is dried in a tower by use of 93% acid and then fed to a four-stage converter. Here the sulphur dioxide is converted to sulphur trioxide over a vanadium pentoxide catalyst and subsequent absorption of sulphur trioxide is carried out in two towers which produce 98% sulphuric acid and 20% oleum, respectively.

ITALY

Fourth World Petroleum Congress

A provisional programme has been issued for the Fourth World Petroleum Congress, which will be held in Rome from June 6-15, 1955. Four speakers of international repute will give lectures on petroleum subjects of general interest during the Congress. Sections will be formed to cover the following subjects:

(1) Geology and geophysics.
(2) Drilling and production.
(3) Oil processing.
(4) Production of chemicals from petroleum. Properties and application.

(5) Composition of petroleum. Analysis and testing. Measurement and control.

(6) Utilisation of oil products.

(7) Construction of equipment. Corrosion. Materials.

(8) Transport, storage and distribution.

(9) Economics, statistics, education and training.

The possibility of organising an exhibition of petroleum industry on the occasion of the Congress is being considered. The arrangements include a number of visits of both industrial and tourist interest.

The address of the General Organising Committee is Via Tevere 20, Rome.

PORTUGAL

Fertiliser plans

The Government's long-range plan for the chemical fertiliser industry envisages investment of 1,080 million escudos (£13,500,000) in order to produce annually 9,000 tons of calcium cyanamide, 140,000 tons of sulphate of ammonia and 80,000 tons of nitrates. The first phase will cost 153 million escudos and comprise:

(1) Installing another electric furnace in the Canas de Senhorim cyanamide plant of the Companhia Portuguesa de Fornos Electricos (Largo do Directorio 4-2°, Lisbon). Cost: 15 million escudos.

(2) Enlarging the Estarreja factory of Amoniaco Portugues (Rua Victor Cordon 19-1°, Lisbon) to produce 70,000 tons of sulphate of ammonia p.a. Cost: 120 million escudos.

(3) Enlarging the Barreiro sulphonating plant of the Uniao Fabril de Azoto (Rua da Prata 25c-20-D, Lisbon) to cope with the planned increase in ammonia production. Cost: 18 million escudos.

A subsequent phase will include the construction of a new nitrate factory (to produce 35,000 tons of ammonia p.a. as material for the new nitrate production and the increased sulphate of ammonia production) and the re-equipment of the Uniao Fabril de Azoto's Alferrarede factory (for a higher production of ammonia to be sulphonated at Barreiro).

EGYPT

Aswan fertiliser project

The National Production Council recently invited 11 foreign firms (including one in the United Kingdom) to bid for the construction and management of a fertiliser plant at Aswan to produce 370,000 tons of ammonium nitrate 20.5%, based on electric current generated at the Aswan Dam hydroelectric station now under construction. The completed project is now estimated to cost about £25 million, of which about £15 million would be in foreign exchange to pay for the machinery, etc. Experts who have examined the project on behalf of the Egyptian Government are definitely in favour of its execution. Egypt is at

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present highly dependent upon imports of fertiliser (526,000 tons were imported last year), and for that reason alone the erection of the Aswan plant is justifiable. In addition, it also appears likely that the plant will be an economic proposition.

HUNGARY

New synthetic fibre

A new synthetic material, *Efylon*, which has been made up into garments such as pullovers and socks with the appearance of wool, is stated to be several times more durable than nylon. The new material is said to be produced by chemical treatment of waste synthetic thread of nylon, *Orlon*, *Dacron*, *Capron* and *Perlon*. Clothes made of the material are said to have been given heavy wear for six months without producing a break.

West German chemical exports

West German exports of chemical goods totalled 1,380 million marks in the first half of this year, about 9% more than in the second half of last year, according to industry sources. The rate of increase of sales abroad slowed down considerably in the last few months, owing to increased competition on the world market, trade restrictions and other difficulties. Exports to the United States, especially sales of staple fibres and tar products, showed the strongest decline.

Chemical exports in the first half of this year accounted for about 22% of the industry's overall sales.

The rise in the plastics production did not continue in the first half of this year to the same extent as in previous years. The output of plastic raw materials in the first half of this year reached 147,000 tons, about 12% more than in the second half of 1953. Exports of plastic raw materials amounted to 21,000 tons in the first half of this year, almost 15% more than in the second half of 1953. They accounted for 7% of all chemical exports.

Bitumen plant

Various grades of bitumen required by the German market are now being produced at Anglo-Iranian's B.P. refinery at Hamburg, where a bitumen plant with a capacity of 50,000 tons p.a. has been commissioned. The new plant uses as feedstock residue oil from the vacuum distillation unit commissioned earlier this year.

Adjacent to the plant is barrelling and other ancillary equipment. The bitumen will be distributed in bulk in rail and road tankers, and also in drums, blocks and sacks.

JORDAN

Industry and mining

The new cement factory at Fuheis was opened in February and is already producing sufficient cement to meet Jordan's requirements at a more economic price than that of imported cement.

A British firm of consultants has completed its survey of the phosphate, manganese and copper deposits at Al Hasa and Wadi Dana. They reported that the phosphate deposits at Ruseifa were large and of good quality, but those at Al Hasa were disappointing. The manganese contained a large percentage of copper and the possibility of exploiting this seemed remote. Further investigation into this and the possibilities of the copper were necessary.

Under an agreement made between a Jordan phosphate mining company, the Development Board and a Belgium firm, the latter will supply £800,000-worth of railway and mining equipment, including 30 railway wagons and other rolling stock mainly for transporting phosphates.

Exports of phosphates during the first half of the year totalled 25,300 tons.

Little progress was made in the investigations into establishing a potash industry.

NETHERLANDS

New soda company

The N.V. Nederlandse Soda Industrie (Netherlands Soda Industry Ltd.) has been officially established at Amsterdam with a capital of 30 million guilders.

The new company will build a big works at Delfzijl with an estimated annual output of 80,000 tons, of which 40,000 tons will be for export. The soda will be made from big salt deposits at Winschoten, near Groningen.

Chemical exports

The annual report of the Netherlands Chemical Industry Association shows that Dutch exports of chemical products and raw materials exceeded imports in value in 1953 but not in volume. Over half the Netherlands exports and over 60% of her imports of chemical products are destined for or come from European countries.

SWEDEN

Aluminium expansion

The Swedish Aluminium Co. is to increase the output capacity of its Sundsvall works from 10,000 to 13,000 tons. Together with 2,000 tons annually produced electrolytically in Dalarna, the company will now be able to meet more than half the total domestic demand, which in 1953 amounted to about 27,000 tons.

DOMINICAN REPUBLIC

Furfural from bagasse

On the occasion of a visit by Mr. Henry D. du Pont of the American firm of E. I. du Pont de Nemours & Co. to the new furfural plant which is being constructed at La Romana, it was announced in the Press that his firm would be the principal buyers. It was further reported that they would be prepared to pay higher prices than they are already paying in the U.S.A. in order to help the new industry, which is claimed to be the first in the world designed to extract furfural from bagasse on a commercial scale.

In accordance with the general policy of the Dominican Government of helping new industries, the operating company, Central Romana By-Product Co. Inc., will be exempt from all taxes, except profits tax, during the next 20 years. This exemption will also apply to customs duties on machinery and on oil which will have to be imported to replace bagasse, which has formerly been used as fuel in the mill and also in the sugar fields. From a total of approximately 195,000 tons of bagasse, it has been estimated that it will be possible to extract about 13,000 tons of furfural.

Cement factory expansion

National capacity for the production of cement was doubled recently by the commissioning of a new furnace, mill and cooler at the factory of Fabrica Dominicana de Cemento in Ciudad Trujillo. The new equipment is capable of producing 12,000 bags/day of cement. Production of cement in the Dominican Republic was started in 1947 with 11,500 metric tons, and 1953 production amounted to 108,000 metric tons.

The manufacturers of the furnace and the mill were the Allis Chalmers Manufacturing Co., of Milwaukee, U.S.A., and the cooler was supplied by the Fuller Co., of Philadelphia. The value of the equipment was reported to be R.D. $619,000, and the cost of freight, installation and other works brought the total expenditure to R.D. $1 million.

JAPAN

Salt industry contemplated

Two Japanese Government representatives have been in India to study Indian salt production methods. Japan is at present importing salt from India. The Japanese Government hopes that as a result of this visit Japan will be able to produce salt on a large scale and so satisfy her requirements herself.

MEXICO

Chemical pulp plant

Celulosa de Chihuahua S.A., with an investment of 200 million pesos, should shortly come into production at Bustillos, Chihuahua. Capacity is for 25,000 tons p.a. of bleached and semi-bleached chemical pulp for writing and printing paper and 7,000 tons of cellulose for artificial fibres. With the operation of this plant over 50% of present imports of chemical pulp will be avoided.

YUGOSLAVIA

Cement factory for Trieste

Work is to begin shortly on a cement factory, with an annual capacity of 150,000 tons, which is to be built at Umago, Trieste. The plant is expected to go into production in 1957, and 80% of its output will be exported. Building costs will amount to some 1,500 million dinars, which has already been allocated by the Yugoslav Federal Government.

Caustic soda production

Yugoslav production of caustic soda, which is one of the country's most important exports, rose from 14,513 tons in 1938 to 22,545 tons in 1953, according to the Yugoslav Press Agency. Total output is expected to reach 23,000 tons in 1954 and 35,000 tons in 1955.

Yugoslav exports of caustic soda have also increased, rising from 7,582 tons in 1939 to 14,045 tons in 1952 and 13,337 tons in 1953.

Whereas Yugoslavia's main outlet before the war was Hungary, with smaller quantities going to Albania and Yugoslavia, recipients now total twelve countries, of which seven are in Europe, four in the Near East and one in South America. The main destinations in 1953 were Austria with 7,644 tons, the Netherlands with 949.7, Turkey with 1,068.9, Switzerland with 1,199.1, Egypt with 844.1 and Brazil with 926.4 tons.

UNITED STATES

Filtration-extraction of oilseeds

The first 13,300 tons of Delta cottonseed processed in a 150-tons/day ' package plant ' by the filtration-extraction process at Greenwood, Mississippi, have shown substantial increase in operating profits, it is reported. This gain has averaged more than $750 per day during some 30 days of continuous specification performance.

The filtration-extraction process, described in CHEMICAL & PROCESS ENGINEERING, 1953, **34** (12), 374, is based on the original solvent process studies conducted by the Southern Regional Research Laboratory of the United States Department of Agriculture. The Greenwood plant, which was designed and engineered by the Lukenweld division of Lukens Steel Co., Coatesville, Pa., is claimed to be the first of its type in the world which can alternately process either cottonseed or soya beans directly to produce high-quality oil and meal.

The oil produced is claimed to be of high quality, with an average gossypol content of 0.053%. No reversion occurred in storage, according to official laboratory reports covering periodic tests and shipments. The meal has a free gossypol content varying between 0.04 and 0.05%, and has commanded a premium as a protein supplement.

The unground meal is relatively low in dust content. Finished meal will be further improved by a new grinding system now being installed. This will yield a meal texture expected to be comparable to that produced with the hydraulic process.

Electricity required to operate the plant is 21.6 kwh./ton, and 700 lb./ton of steam and 2,300 gal./ton 71°F. of water are used in preparation and extraction. Steam is used at a rate of 4,400 lb./hr. and water at 244 gal./min.

The average solvent loss during specification operation amounted to 1.45 gal./ton. While this is lower than that of a direct solvent extraction plant, it is somewhat higher than the lowest figure reported for a prepress plant. When planned changes to equipment seals are completed during the summer shutdown, it is expected that solvent losses will be reduced to about 1 gal./ton.

Uranium from phosphate rock

A new $15-million phosphate chemicals plant which has started operation near Bartow, Florida, U.S.A., is producing as a by-product uranium compounds for the U.S. Atomic Energy Commission. The uranium compounds are recovered as an intermediate step in the production of multiple superphosphate and dicalcium phosphate. A production of 100,000 tons p.a. of dicalcium phosphate is anticipated.

The Bartow plant is the first of its kind in Florida. However, two other companies, the Blockson Chemical Co. of Joliet, Illinois, and Texas City Chemicals Inc., are also producing uranium from Florida-mined phosphate rock, in plants outside

★ **Mr. Donald McDonald,** chairman of the executive committee of the board of Johnson, Matthey & Co. Ltd. since 1950, has retired after 44 years' service with the company. He joined the company direct from University College, London, in 1910 as a junior chemist (having gained his B.Sc. with first-class honours) and as early as 1920 was appointed manager of the refineries at Hatton Garden. He assisted in the design and construction of the new electrolytic silver refinery, and later turned his attention to the development of the chlorine process of gold refining and the installation of the necessary equipment. In 1939 he was appointed to the board as director of the bullion department and of research, adding to his duties in ensuing years the directorship of labour management and mechanical production. Mr. McDonald has published an authoritative account of the life of the founder of the company, Percival Norton Johnson, and has recently been engaged upon the compilation of a detailed history of platinum from its discovery until the 1880s. At a brief ceremony in the recently completed Hatton Garden office block—in the construction of which he was closely associated. —Mr. McDonald was presented with a cheque and a bound book of signatures of his colleagues and friends throughout the company.

★ **Dr. G. H. Beeby** has been appointed chairman of the Chemical Divisional Council of the British Standards Institution and has, therefore, at the same time become a member of the General Council and the Executive Committee. Dr. Beeby is chairman of I.C.I. Salt Division at Winsford in Cheshire and has spent all his working life in that organisation. Born in Leicestershire and educated at Loughborough College and London University, he joined the Billingham Division of I.C.I. as a chemist in 1928. Subsequently he spent 13 years in I.C.I. sales offices before taking up his present duties in 1952. He is no stranger to the B.S.I., for he first joined the Heavy Chemicals Industry Committee in 1945, and has been its chairman, as well as chairman of the B.S.I. Sub-Committee for international work on chemistry, since 1948.

★ **Mr. C. A. O. Rideal,** director of sales, May & Baker Ltd., recently completed 25 years' service with the company and on

Mr. Donald McDonald.

his return from a business trip to the Americas was presented with a gold watch by the managing director, Dr. T. B. Maxwell, to mark the occasion. Mr. Rideal joined the company at Battersea in 1929. In 1939 he was made commercial manager and two years later he was appointed to the board of directors.

★ The directors of Davey, Paxman & Co. Ltd. announce with regret that for personal reasons **Sir John Greaves** has decided to relinquish his position as managing director of the company and, although he continues as a director of Ruston & Hornsby Ltd., he retires from the board of Davey, Paxman & Co. Ltd.

Mr. G. W. Bone, assistant managing director, has been appointed to succeed Sir John as managing director.

★ We regret to announce the death, at the age of 60, of **Mr. F. W. Woodfield,** manager of the Manchester office of Babcock & Wilcox Ltd. Mr. Woodfield, who was well known in engineering circles, was a past chairman and member of the Technical Advisory Committee for the Fuel Efficiency Exhibition and an active member of the Engineers Club. He joined Babcock & Wilcox in 1929 and was appointed to the Manchester Office in 1935. He was for many years a director of Erith's Engineering Co. Ltd.

the State. Meanwhile, a fourth company, Virginia-Carolina Chemical Corporation, is expected to begin making uranium at Nichols, Florida, shortly.

At present the U.S. has to import a major portion of its uranium supplies. The principal foreign sources are Canada, the Belgian Congo and South Africa. Australia is a promising potential source. Domestic production at present is in the

Colorado plateau area where there are a number of mills which concentrate and extract uranium from carnatite-roscoelite-type vanadium-uranium ores and other ore types as sufficient reserves are developed. We reported in our September issue that the Uranium Reduction Co. is planning to construct a mill at Moab, Utah, to treat uranium ores from Utah and Colorado areas.

Larger fertiliser plants?

A possible pattern for technological changes in the fertiliser industry was outlined before a recent meeting of the American Institute of Chemical Engineers in Washington. These changes could involve a shift from the conventional small fertiliser plant of about 50,000 tons annual capacity to an integrated continuous-process plant producing at the rate of several hundred thousand tons per year.

It is questionable whether the conventional small fertiliser plant will continue to be the most economical unit for meeting the greatly increasing demand for fertilisers. During the last 20 years the annual consumption of fertilisers in the United States has risen from 5 million to 23 million tons. Over the next 20 years, with an expected population increase of 40 million and with farm acreage expected to remain fairly constant, the need for greater per acre production of food should continue the sharp increase in fertiliser consumption.

Secondly, cost-conscious consumers have focused attention on production cost, analysis and properties of fertilisers. To meet present and future demands, therefore, manufacturers need to produce from the cheapest possible raw materials, by the most flexible process, and at low operating cost. Such production can probably be achieved best through the modern chemical process industry, which has made available new cheap raw materials and a variety of continuous processes adaptable to the fertiliser industry.

Mr. R. B. Filbert, who presented the paper to the chemical engineers, stressed that it was not possible, at this time, to say how extensively continuous-processing might take hold of the fertiliser industry. He said that because of the many assumptions required, nothing short of a detailed study of a particular manufacturer's operations and his regional market can furnish a reliable estimate of the minimum or maximum investment in process improvement that will yield him an attractive return.

PHILIPPINES

New industries

The Caltex oil group has completed a refinery at Batangas Bay, 196 km. south of Manila, with a capacity of 13,000 bbl./day of crude oil.

Plans have been announced for the establishment of a metals reduction plant and of factories to produce aluminium products (7 million lb. estimated capacity during the first year), two for the production of explosives and others to produce antibiotics, pharmaceuticals, printing ink and a pilot plant for the extraction of castor oil.

The establishment of new industries in the country is being actively encouraged by the Government which offers exemption from taxes and import duties and the provision of foreign exchange to those enterprises for which the Government's prior approval is given.

SOUTH AFRICA

Cheap ilmenite process

For several months the Titanium Corporation of S.A. Ltd., of Umgababa, Natal, have worked deposits on the Natal south coast which proved to contain an estimated 2 million tons of ilmenite, 200,000 tons of zircon and 100,000 tons of rutile. The directors of the corporation believe they are mining ilmenite more cheaply than any other country in the world, producing and delivering it in New York at lower prices than the Americans can produce from their own deposits. The corporation has designed its own electro-magnetic plant, claimed to be the latest of its kind in the world, to separate the ilmenite from the zircon and rutile in a single operation. It can produce 300 long tons of ilmenite a day.

AUSTRALIA

Uranium production plans

South Australia's A£5-million uranium mine and concentration plant at Radium Hill is due to begin full production shortly, and the first exports of high-grade uranium oxide to Britain and the United States are expected to be shipped early next year.

The State Premier, announcing this, added that uranium concentrates would have to be stockpiled until the new refinery at Port Pirie is ready early in 1955.

PAKISTAN

Sulphuric acid

The sulphuric acid plant now under construction at Lyallpur is expected to go into production by the beginning of August. The machinery was originally imported by a private business firm, but has since been taken over by the Pakistan Industrial Development Corporation. The Pakistan Industrial Development Corporation is also planning a superphosphates plant with a production capacity of 20 tons/day at Lyallpur. Work on this project is expected to begin shortly.

MEETINGS

Chemical Society

October 14. 'Chemical Research at Harwell,' by Dr. R. Spence, 7.30 p.m., North British Station Hotel, Edinburgh. Joint meeting with the R.I.C. and the S.C.I.

October 14. 'The Production and Properties of Titanium and Titanium Alloys,' by Dr. N. P. Inglis, 7.30 p.m., Agriculture Lecture Theatre, Queen's University, Belfast. Joint meeting with the R.I.C. and the S.C.I.

October 18. 'The Stereochemistry of Catalytic Hydrogenation,' by Prof. R. P. Linstead, 5 p.m., University College, Leicester.

October 22. 'Gas - Liquid Chromatography,' by Prof. A. Robertson, 8.30 p.m., Chemical Laboratory, the University, Cambridge.

October 28. 'Some Recent Advances in Fluorine Chemistry,' by Prof. H. J. Emeleus, 7 p.m., Department of Chemistry, the University, Bristol. Joint meeting with the R.I.C. and the S.C.I.

October 28. 'Chemisorption and Catalysis,' by Dr. F. C. Tompkins, 5 p.m., Chemistry Lecture Theatre, the University, Liverpool. Joint meeting with the R.I.C., S.C.I. and the British Association of Chemists.

November 1. 'The Polymerisation of Sulphur,' by Prof. G. Gee, 7.45 p.m., Chemistry Lecture Theatre, Queen's University, Belfast. Joint meeting with the R.I.C. and the S.C.I.

November 4. 'On Open and Closed Sequences in Reaction Kinetics,' by Prof. J. A. Christiansen, 7.30 p.m., Large Chemistry Lecture Theatre, Imperial College of Science and Technology, South Kensington, S.W.7.

Society of Chemical Industry

October 19. 'Recent Investigations in the Field of Vinyl Polymerisation,' by Dr.

C. H. Bamford, 6.30 p.m., rooms of the Chemical Society, Burlington House, Piccadilly, London, W.1.

Institute of Fuel

October 8. 'Desulphurisation of Coke-oven Gas by Selective Quick Washing with Ammonia Solution and the Conversion of Hydrogen Sulphide to Sulphur, Sulphuric Acid or Ammonium Sulphate,' by Dr. H. Bähr, 7 p.m., North British Hotel, Edinburgh. Joint meeting with the Institute of Petroleum.

October 14. 'Heat Transfer in a Continuous Reheating Furnace,' by Prof. R. J. Sarjant and Dr. D. Smith; and 'The Significance of Sectional Heat Balances and Burner Performance in Furnaces,' by Dr. N. P. Bacon and Prof. R. J. Sarjant; 5.30 p.m., Institution of Civil Engineers, Great George Street, London, S.W.1.

Incorporated Plant Engineers

October 20. 'Recent Research in Pressure Vessels,' by P. H. R. Lane, 7 p.m., Bull Hotel, Rochester, Kent.

Institute of Petroleum

October 20. 'Platforming,' by G. Egloff, 5.30 p.m., Chemical Society, Burlington House, Piccadilly, London, W.1.

Export Opportunities

Chemicals

Senor Migual Nunez del Prado of Casilla Postal 592, Arequipa, Peru, wishes to represent United Kingdom manufacturers of the following chemicals: tartaric, citric, and formic acids, sodium sulphate anhydrous, sodium arsenate, sodium phosphate, caustic soda, paraffin wax, and chemicals for tanning leather.

Senor del Prado wishes to act as a commission agent for the above goods, some of which he might occasionally purchase for his own account. Payment will be made on a cash against documents basis.

Firms interested in this enquiry should *write in Spanish* direct to the above address, giving full details of their offers. (Board of Trade reference: ESB/17560/54.)

Proveedora Industrial S.A., of V. Carranza, 859, Apartado Postal 462, Mexico D.F., who deal with the import, sale and distribution of industrial products generally, have asked the British Embassy at Mexico City for assistance in obtaining agencies from United Kingdom manufacturers of cellulose, rubber accelerators, citric acid, caffeine, sodium sulphite, sodium sulphide, fruit essences, sulphides, chlorides, sodium potassium, arseniates (various).

Manufacturers interested in this enquiry should write direct to the Mexican firm, by air mail, giving full particulars of their products and quoting prices c.i.f. in dollars. (Board of Trade reference: ESB/17352/54.)